Analytical Abstracts

A monthly publication dealing with all branches of analytical chemistry: issued by the Society for Analytical Chemistry

Volume 8
No. 8, Abstracts 3141 - 3559

August, 1961

Published for the Society by
W. HEFFER & SONS LTD., CAMBRIDGE, ENGLAND

Binding . . .

W. Heffer & Sons Ltd. announce that they are now able to BIND copies of ANALYTICAL ABSTRACTS, in an approved binding case, at a cost of 14/6 per volume. The 12 Parts for 1960 together with Index and remittance for 14/6 should be sent to

W. HEFFER & SONS, LIMITED
HILLS ROAD, CAMBRIDGE

IMPORTANT NOTICE TO SUBSCRIBERS

(Other than Members of the Society)

The Subscription to *Analytical Abstracts*, inclusive of Index, is £5 per annum; and for *Analytical Abstracts* together with *The Analyst* and Indexes is £8 8s. per annum.

The price of single copies of *Analytical Abstracts*, including back numbers, is 10s. 6d. post free.

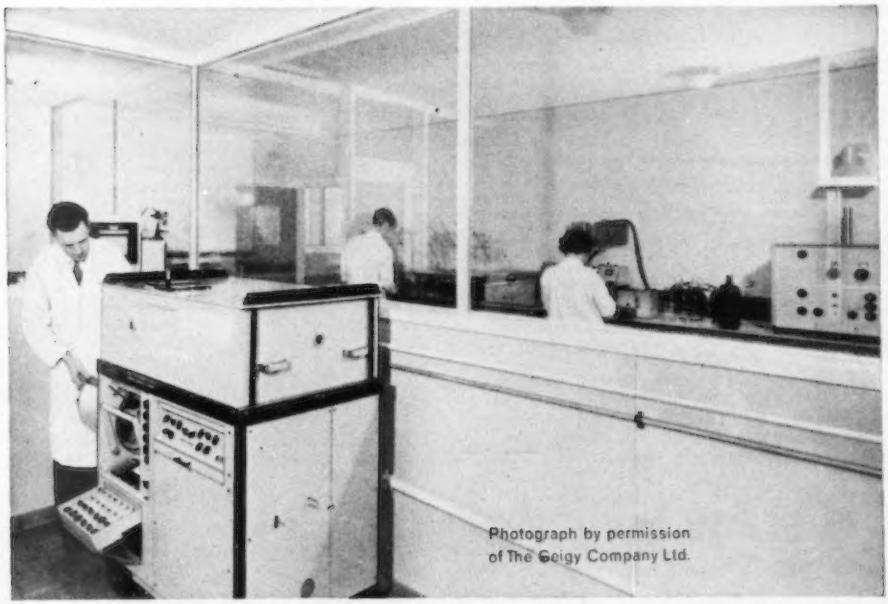
Analytical Abstracts is available printed on one side of the paper only, suitable for incorporation into reference systems; the cost is £5 7s. 6d. per annum, exclusive of Index.

All further enquiries about subscriptions should be made through the Secretary, the Society for Analytical Chemistry, 14, Belgrave Square, London, S.W.1. Telephone: BELgravia 3258.

UNICAM

The highest levels of accuracy and reliability are essential to the quality control service of the pharmaceutical industry. The Unicam SP.100 Infrared Spectrophotometer provides more information in less time and with less effort—to performance levels previously associated only with research equipment built to specialist specification.
Detailed attention to operating systems and a comprehensive monitoring routine allow even the unskilled operator to achieve the outstanding performance offered by the SP.100. For information on how the SP.100 can help in your laboratory please write to: Unicam Instruments Limited, York Street, Cambridge.

S.P.100 Infrared Spectrophotometer

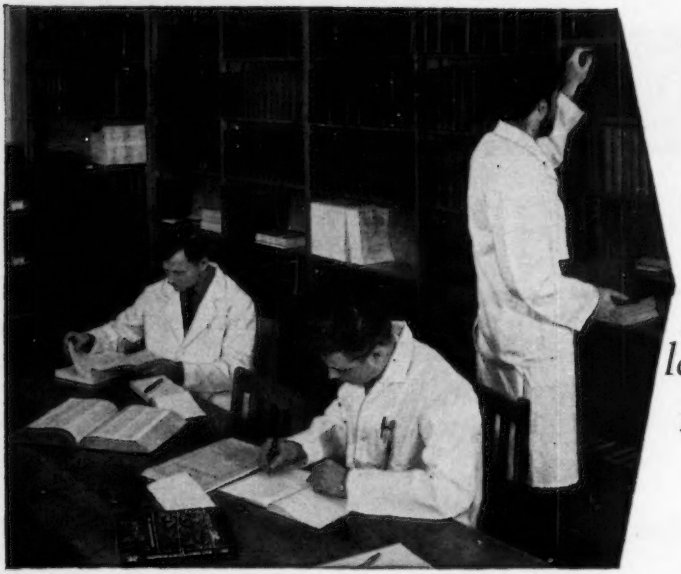


Other Unicam instruments appearing in this photograph are the SP.500 Ultraviolet Visible and Near Infrared Spectrophotometer and the SP.900 Flame Spectrophotometer.

UNICAM INSTRUMENTS LIMITED · YORK STREET · CAMBRIDGE

Incidental information

No. 15



Items of interest from our laboratory notebooks

Acid radicles by colorimetry

This method, depending on the liberation from chloranilates of an equivalent amount of highly coloured chloranilic acid, is continually gaining in scope as the appropriate chloranilates become available.

We now prepare and supply from stock:—

Barium chloranilate for **sulphate**
(J. E. Barney and R. J. Bertolacini, *Anal. Chem.* **30**, 202 (1958)).
CODE 1909 Price—5g: 10/9, 10g: 20/-

Mercuric chloranilate for **chloride**
(the above paper and *idem, ibid.* **29**, 1187).
CODE 5541 Price—5g: 10/6, 10g: 19/6

Lanthanum chloranilate for **fluoride**
(K. Hayashi *et al., Talanta*, **4**, 126 (1960)) and for **phosphate** (*idem, ibid.*, **4**, 244 (1960)).
CODE 5002·5 Price—5g: 11/9, 10g: 22/-

For analysis and production

1:10-Phenanthroline, a sensitive reagent that gives a stable colour with ferrous ions, can be used to mask Cd^{2+}, Co^{2+}, Cu^{2+}, Ni^{2+}, Mn^{2+} and Zn^{2+} in the complexometric titration of Pb^{2+} and Al^{3+} with EDTA. (R. Pribil and F. Vydra, *Coll. Czech. Chem. Commun.*, **24**, 3103 (1959) (in German): *Anal. Abs.* 2668 (1960)). It is also a **dryer-catalyst** and, in conjunction with manganese, can shorten the drying time of printing ink on 'acid' papers (W. H. Canty *et al., Ind. Eng. Chem.*, **52**, 67 (1960)). We offer in bulk for industrial use at considerably reduced prices.
CODE 6580 Price—1g: 6/-, 5g: 23/3

Greater selectivity

As the metal indicator Xylenol Orange is used in acid solution, the EDTA titration is unaffected by some common metal ions that do not form EDTA complexes at low pH. Selectivity can be increased still further by using **acetylacetone**, which will mask Fe^{3+}, Al^{3+}, Be^{2+}, Pd^{2+} and UO_2^{2+} in, for instance, the determination of Zn^{2+} or Pb^{2+} (W. Z. Jablonski and E. A. Johnson, *Analyst*, **85**, 297 (1960)).

The above are further examples from the great range of up-to-date reagents made available by Hopkin & Williams Ltd.

H&W
TRADE MARK

CHEMICALS

for research, analysis and industry

HOPKIN & WILLIAMS LTD., CHADWELL HEATH, ESSEX, ENGLAND
Branches: London, Manchester, Glasgow *Agents throughout U.K. and all over the world*
TAS/HW.14

ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

General reviews of progress, reagents and methods of general application.

3141. **Classification and nomenclature of electrochemical methods of analysis.** P. Delahay, G. Charlot and H. A. Laitinen (Dept. of Chem., Louisiana State Univ., U.S.A.). *J. Electroanal. Chem.*, 1960, **1** (6), 425–433 (in French).—A rational classification and nomenclature of electrochemical analytical methods is proposed. Methods based on electrokinetic phenomena are excluded. N. E.

3142. **X-ray spectroscopy applied to industry.** Proceedings of an International Conference, Liège, June 1960, organised by Ass. des Ingén. Diplomés par l'Univ. de Liège. *Rev. Univ. Min.*, 1961, **17** (4), 143–301.—The following papers are included. **Possibilities and limitations of analysis by X-ray spectrography,** A. Guinier, 143–150 (in French). **A new ARL instrument for X-ray fluorescence,** E. Davidson, W. H. Barry, R. Capua and J. M. Carrol, 150–158 (in French). **Position and evolution of apparatus for X-ray spectrometry,** R. Griffoul, 158–164 (in French). **Fundamental, preparative and sample-size limits in X-ray fluorescence analysis,** H. Neff, 164–173 (in German). **Development and evolution of X-ray spectrometers of the Philips Group intended for elementary analysis by X-ray fluorescence,** J. L. de Vries, 173–176 (in French). **A new automatic X-ray fluorescence spectrometer,** J. R. Stansfield, 177–180 (in English). **Description of the Solartron vacuum automatic X-ray spectrometer and programme selection by punched card,** J. Lucas-Tooth, 180–183 (in English). **A.R.L. - B.I.S.R.A. calculator for absorption effects in X-ray fluorescence analysis,** D. F. Sermin and R. H. Tyas, 183–189 (in English). **Comparison of the practice of X-ray and optical spectrometry for the analysis of ordinary and special steel,** R. Loude and R. Billet, 190–195 (in French). **Applications and analytical results with A.R.L. X-ray instruments,** R. H. Tyas, 195–203 (in English). **Application of X-ray fluorescence analysis to the field of light metals,** H. Pfundt, 203–207 (in German). **Application of X-ray fluorescence to analytical control in a steel-mill,** R. Berger and P. Deceuleneer, 207–214 (in French). **The introduction of X-ray fluorescence analysis in the ironworks industry,** H.-J. Kopineck, 214–223 (in German). **X-ray fluorescence spectroscopy in metallurgical research,** T. A. Davies, 228–239 (in English). **Absorption and emission methods of X-ray micro-analysis,** P. Duncumb, 240–246 (in English). **Metallurgical applications of the X-ray scanning micro-analyser in an industrial laboratory,** D. A. Melford, 247–252 (in English). **Metallurgical applications of the micro-probe of Castaing,** J. Philibert, 252–256 (in French). **Determination of copper, lead and zinc in roasted ores and flue dust,** M. Herrmann, 257–259 (in German). **Application to chemical analysis of X-ray excitation by rays emitted by radioactive sources,** G. Seibel, J. Y. Le Traon and P. Martinelli, 260–275 (in French). **Some applications of X-ray fluorescence spectrography in the petroleum industry,** G. Baron and C. Fagot, 275–279 (in French). **Determination of traces of metals in organic products by means of X-ray fluorescence spectrography,** R. Westrik, 279–282 (in English). **Statistical study of the results obtained by X-ray fluorescence and the electrochemical method in the determination of silver in photographic materials,** L. J. Moelants and Z. Hainski, 282–291 (in French). **Some applications of X-ray spectrometry to the analysis of various products,** R. Rabillon, 291–298 (in French). **Precise and rapid determination of uranium in its compounds by X-ray fluorescence—application to uranium carbides,** R. Tertian, F. Gallin and R. Geninasca, 298–301 (in French).

3143. **Precision and limit of detection of analytical methods.** A. L. Wilson (Central Electricity Res. Lab., Kingston Rd., Leatherhead, Surrey, England). *Analyst*, 1961, **86**, 72–74.—Definitions of the limits of detection and the precision of analytical methods are given. It is pointed out that more attention should be paid to the standard deviation of the blank value in determining the parameters. Some aspects of the effect of change in precision with change in response are considered. B. J. WALBY

3144. **Analysis for industry.** A. M. G. Macdonald. *Ind. Chem.*, 1961, **37**, 30–32.—The application of cupferron analogues and derivatives is discussed. It is concluded that, in general, few of the suggested reagents possess advantages over those formerly used and, of the hydroxylamines, only *N*-phenylhydroxylamine appears promising; *N*-furoylbenzoylhydroxylamine may repay further investigation and the results obtained with *N*-phenylazohydroxylamine require confirmation. It is considered that phenylhydroxylamine derivatives give better results than hydroxamic acid, but the introduction of other groups might well yield better reagents, although lack of specificity will always be a drawback. (19 references.) S.C.I. ABSTR.

3145. **Thenoyltrifluoroacetone [TTA]: polarographic and spectrophotometric behaviour, and dissociation equilibria. Mechanisms of electrolytic reduction.** P. J. Elving and P. G. Grodzka (Univ. Michigan, Ann Arbor, U.S.A.). *Anal. Chem.*, 1961, **33** (1), 2–11.—TTA gives four polarographic waves whose presence and properties depend on the pH, the buffer used and its concn. Spectrophotometric examination of TTA soln. before and after reduction has explained the complex polarographic behaviour in terms of the various tautomeric and acid - base equilibria involving TTA species. G. P. COOK

3146. **Deactivation of oxidation layers in an atmosphere of oxygen.** G. Kainz and H. Horwatitsch (Anal. Inst., Univ., Vienna). *Z. anal.*

Chem., 1961, **178** (6), 406–411 (in German).—The efficiency of oxidation reagents for the burning of, for example, methane, decreases with a preliminary heating in oxygen. The variation of activity with temperature is reported for 5 forms of CuO, for 3 forms of Co_3O_4, for Cr_2O_3, and for MnO_2. This decrease in activity is attributed to partial sintering of the oxides. T. R. ANDREW

3147. **The preparation of the Luff - Schoorl copper reagent.** A. J. Weide and E. L. Krugers Dagneaux (Lab. N.V.P. de Gruyter & Zn, 's-Hertogenbosch, Netherlands). *Chem. Weekbl.*, 1961, **57** (4), 46–47.—The reagent as originally prepared by Schoorl (*Ibid.*, 1912, **9**, 678) is difficult to standardise. A modification has been made so that a constant amount of HCO_3^- remains in the soln., and the pH is 9·9. Highly reproducible results are attained: the reduction value varies from 99·5% to 100·5%. *Procedure*—Dissolve 125 g of $Na_2CO_3.10H_2O$ in 110 ml of hot H_2O, cool and mix carefully with a soln. of 50 g of citric acid in 50 ml of H_2O. Swirl the soln. until CO_2 evolution has ceased, then add to a cooled soln. of 263 g of $Na_2CO_3.10H_2O$ in 250 ml of hot H_2O. Add a soln. of 25 g of $CuSO_4.5H_2O$ in 100 ml of H_2O and make up to 1 litre with H_2O. The pH must be 9·9. M. J. MAURICE

3148. **Contributions to organic spot-test analysis.** F. Feigl, D. Goldstein and D. Haguenauer-Castro (Lab. da Prod. Mineral, Min. da Agric., Rio de Janeiro, Brazil). *Z. anal. Chem.*, 1961, **178** (6), 419–428 (in English).—Spot tests are described for the detection of hexamine, benzoyl peroxide, triphenyl-phosphine, -arsine, -stibine and -bismuthine, mucic acid, cyclohexylsulphamic acid, hippuric acid, anthraquinone and anthraquinone dyestuffs. T. R. ANDREW

3149. **Tervalent molybdenum as volumetric reducing agent for mixed oxidising agents.** S. Z. Mikhail and J. J. Stewart (Dept. of Nucl. Engng, Kansas State Univ., Manhattan, U.S.A.). *Z. anal. Chem.*, 1961, **178** (5), 335–345 (in English).—The potentiometric titration of binary and ternary mixtures of oxidising agents, namely $Ce(SO_4)_2$, $K_2Cr_2O_7$, NH_4VO_3 and $FeCl_3$ (prep. described), was carried out in HCl or H_2SO_4 media with fresh soln. of Mo^{III} (prep. described) at 85°. Platinum and calomel electrodes were used. The results of this study are discussed and it is shown that, although admixtures of two ions could be accurately determined in some cases, the determination of mixtures of three ions was not satisfactory. B. B. BAUMINGER

See also Abstracts—**3169**, Use of respropiophenone oxime as reagent for Cu. **3279**, Use of formaldoxime as reagent for Fe.

2.—INORGANIC ANALYSIS

General, determination of elements (arranged in the order of the Periodic Table), analysis of minerals and inorganic industrial products.

3150. **The electrolytic micro-determination of metal 8-hydroxyquinolinates.** G. Berraz and O. Delgado (Inst. de Invest., Lab. Invest. Quím. II, Univ. Nac. del Litoral, Santa Fé). *Rev. Fac. Ing. Quím., Argentina*, 1959, **28**, 39–52.—A semi-micro technique for the quant. pptn. of the metal 8-hydroxyquinolinate, which is then dissolved in HCl, is described. The method depends on the bromination of the 8-hydroxyquinoline with bromine generated electrically at constant current. The bromination is carried out in a special cell (illustrated) in which the anode and cathode compartments are separated by a porous glass disc. The cell is also fitted with platinum electrodes to indicate free bromine. Aluminium (20 μg) could be determined with a relative error of ≃1%; a mean deviation of 0·5 μg was obtained in 10 tests with different amounts of Al. B. F. FULLAN

3151. **Determination of trace elements by fast-neutron activation analysis.** R. F. Coleman (U.K. A. E. A., Atomic Weapons Res. Estab., Aldermaston, Berks., England). *Analyst*, 1961, **86**, 39–44.—A method is described for producing a large flux of 14·5-MeV neutrons suitable for activation analysis. With neutrons of such energy many reactions are possible, the most useful being (n, p), (n, α) and $(n, 2n)$. The first two predominate for the lighter elements, but decrease with increasing atomic number. The $(n, 2n)$ cross-section increases with atomic number and, for elements of atomic number >50, is from 1 to 2 barns. The sample is placed as near as possible to the neutron source and for reproducible geometry is either machined or supported in powder form in a plastic holder. Suitable reactions are quoted for most of the elements of the Periodic Table with the cross-section, half-life of the product, probable limits of detection and the elements that may interfere with the expected reaction by forming the same nuclide by a different reaction. A. O. JONES

3152. **Quantitative determination of low-atomic-number elements using intensity ratio of coherent to incoherent scattering of X-rays.** C. W. Diggins, jun. (Petroleum Res. Center, Bur. of Mines, U.S. Dept. of the Interior, Bartlesville, Okla.). *Anal. Chem.*, 1961, **33** (1), 67–70.—The method described has been developed for the determination of elements of atomic number ≯12. Carbon and H may be determined in hydrocarbons, in the presence of additional elements, by quadruplicate experiments in ≯20 min. Conventional X-ray spectrographic equipment has been used without modification, and accuracy equal to that of micro-combustion techniques is claimed. E. G. CUMMINS

3153. **Chloranilic acid as a reagent in the paper chromatography of inorganic compounds. II. Heavier metals.** H. S. R. Barreto, R. C. R. Barreto and I. P. Pinto (Nat. Sch. of Agric., Rural Univ., Rio de Janeiro, Brazil). *J. Chromatography*, 1961, **5** (1), 5–8 (in English).—The acetates of Fe^{3+}, Ni^{2+}, Co^{2+}, Zn^{2+}, Ag^+, Cd^{2+}, Hg^{2+}, Pb^{2+} and U^{4+} can be separated by two-dimensional paper chromatography with acetone - 8N-acetic acid (9:1) and 96% ethanol - 2N-acetic acid (4:1) as solvents. The separated metals are detected with chloranilic acid (*cf.* Barreto and Barreto, *Anal. Abstr.*, 1961, **8**, 926); the sensitivity varies from 0·01 μg for Fe^{3+} to 5 μg for Hg^{2+} and Zn^{2+}. A. R. ROGERS

3154. **Chromatographic micro-determination of copper, nickel, zinc and cadmium.** J. Minczewski and A. Fołdzińska (Inst. of Nucl. Res., Polish Acad. Sci., Warsaw). *Chem. Anal., Warsaw*, 1960, **5** (4) 575–580.—With dithizone-impregnated paper strips (6 mm × 100 mm) buffered at pH 9, 0·025 to 0·25 μg of Cu^{2+}, Cd^{2+}, Ni^{2+} and Zn^{2+} per ml are separated

in that order, which is due to the different solubilities of the pptd. dithizonates. The chromatograms are obtained by dipping a pointed end of the paper strip into a drop of aq. NH_3 containing a micro-drop (0·025 ml) of sample held on the surface of a Petri dish made water-repellent. The separation is clear enough for a semi-quantitative estimation by comparison with standards. Up to 10 μg of Pb, Bi, Fe, Hg, Mn and Sn per ml and 3 μg of Co per ml did not interfere. P. BRYCH

3155. **Chemical analysis of semi-conductor alloys of the system indium - antimony - tellurium.** N. N. Safronkova and Yu. S. Lyalikov (Inst. of Chem., Moldavia Filial of the Acad. Sci., USSR). *Zavod. Lab.*, 1961, **27** (1), 21–22.—The sample (0·25 to 1 g) is fused with six times its wt. of $KHSO_4$, the cooled melt is dissolved in a mixture of 100 ml of water, 50 ml of H_2SO_4 and 50 ml of HCl, and separate aliquots are used for the determinations of Sb, Te and In. For Sb, the soln. (5 to 15 ml) is treated with 5 to 10 ml of HCl (1 : 4) and titrated with 0·1N-$KBrO_3$ in the presence of methyl red indicator. For Te, the soln. is treated with 5 to 10 ml of water and 1 to 2 g of KI, and the iodine liberated is titrated with 0·1N-$Na_2S_2O_3$ in the presence of starch soln. For In, the soln. is treated with 5 ml of 10% Na K tartrate soln., 10 to 15 ml of a buffer soln. (pH 8 to 10) and a small amount of Eriochrome black T indicator, and titrated, while boiling, with 0·01M-EDTA (disodium salt) to the transition from violet to blue. G. S. SMITH

3156. **The formation of hydrogen peroxide in alcohols. Its effect on the extraction of chelates of benzohydroxamic acid.** C. E. Meloan and W. W. Brandt (Purdue Univ., Lafayette, Ind., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 102–104.—The benzohydroxamic acid chelates of U^{VI}, V^{V} and Fe^{III} give low extinction values when they are extracted into an alcohol that has not been freshly distilled; this was found to be due to the destruction of the chelates by traces of H_2O_2 formed in the alcohol. Such formation of H_2O_2 can be avoided by saturating the alcohol with H_2O and storing it in a metal container. The presence of H_2O_2 was detected in n-hexanol and several other saturated alcohols from C_5 to C_{10}. G. P. COOK

3157. **Differential thermal analysis and thermogravimetric analysis of fission-product oxides and nitrates to 1500° C.** P. F. Campbell, M. H. Ortner and C. J. Anderson (Vitro Lab., West Orange, N.J., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 58–61.—The equipment described has been used up to this elevated temperature and new results are given for $CsNO_3$, $Ce(NO_3)_3.6H_2O$, $ZrO(NO_3)_2.2H_2O$ and RuO_2. E. G. CUMMINS

3158. **Separation of parahydrogen, orthohydrogen, hydrogen deuteride, orthodeuterium and paradeuterium by gas-adsorption chromatography.** Takao Kwan (Hokkaido Univ., Sapporo, Japan). *J. Res. Inst. Catalysis, Hokkaido Univ.*, 1960, **8**, 18–28.—A 2-metre spiral copper column packed with 30 to 60-mesh Linde mol. sieve (13A) can be used to separate isotopic and spin isomers at −195°. The column efficiency and retention vol. are sensitive to the water content of the sieve. There is some ortho - para conversion on the column. Rough retention vol. for parahydrogen, orthohydrogen, hydrogen deuteride and deuterium are 3·6, 4·9, 5·0 and 7·0 litres of He, respectively, and for hydrogen deuteride, orthodeuterium and paradeuterium 9·1, 13·2 and 14·4 litres of normal hydrogen, respectively. With He as carrier gas, the separation of deuterium isomers is negligible. The preparation of isotopically pure hydrogen by means of 5A sieve probably produces parahydrogen. These techniques could be used to enrich parahydrogen without using a conversion catalyst and in the absence of a super-refrigerant. CHEM. ABSTR.

3159. **The detection of tritium-labelled compounds in vapour-phase chromatography.** H. E. Dobbs (U.K.A.E.A., Isotope Res. Div., Wantage, Berks.). *J. Chromatography*, 1961, **5** (1), 32–37 (in English).—An ionisation chamber of volume $\simeq$3 ml, operated at a polarising potential of 72 V, is described for the detection of ^3H in a gas flow at temp. <190°. A good response is given by 0·5 μC of ^3H. A. R. ROGERS

3160. **Measurement of the moisture content of gases.** N. I. Shtelnbok. USSR Pat. 132,858 (20.10.1960).—The gas passes through an ionisation chamber, then through a drying agent, and then through a second ionisation chamber; the current differential measures the moisture content of the original gas. The readings are independent of gas composition (provided that only water is absorbed by the drying agent), changes of temp. and pressure, and rate of gas flow. No specific examples are given. C. D. KOPKIN

3161. **Spectrophotometric determination of traces of hydrogen peroxide.** C. E. Meloan, M. Mauck and C. Huffman (Dept. of Chem., State Univ., Manhattan, Kans., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 104–106.—Traces ($>7 \times 10^{-6}$ mole) of H_2O_2 are determined by measuring the decrease in extinction at 380 mμ and 450 mμ, respectively, of the U^{VI} and V^{V} chelates of benzohydroxamic acid in n-hexanol soln. The final hexanol soln. of the chelates contains 1×10^{-5} mole of metal per 10 ml, and is stored in the dark. *Procedure*—Shake the water-immiscible sample with water (15 to 20 ml) at pH 2, and then the aq. fraction with 10 ml of the n-hexanol soln. of the chelate. Water-miscible samples may be shaken directly with the chelate soln. Measure the extinction of the U chelate immediately, but that of the V chelate after 10 min. Alkyl peroxides and hydroperoxides do not interfere. J. P. STERN

3162. **Spectrographic determination of traces of metals in hydrogen peroxide solutions.** K. K. Popkov and L. M. Shtifman. *Zhur. Anal. Khim.*, 1961, **16** (1), 113–114.—Hydrogen peroxide soln. are evaporated on a heated graphite electrode in the form of a spike with a concave hemispherical head. The part of the electrode containing the dry residue is completely burnt in an a.c. arc. Nickel, Fe, Sn, Cr and Al can be determined in H_2O_2 soln. in the range 10 to 1000 mg per litre and Cu in the range 0·2 to 3 mg per litre. Cobalt sulphate is introduced into the peroxide soln. beforehand and the line Co 2580 is used as the internal standard for Al 2568, Fe 2598, Fe 2599, Cr [Cu ?] 2666 and Cr 2677, and Co 3044 is used for Ni 3002, Ni 3003, Sn 3034, Cu 3247 and Al 3092. The relative mean error is 10 to 15%. A. BURWOOD-SMITH

3163. **Determination of argon in air by gas-adsorption chromatography.** Takao Kwan (Hokkaido Univ., Sapporo, Japan). *J. Res. Inst. Catalysis*

Hokkaido Univ., 1960, **8**, 14–17.—Argon, O and N are separated by using H as carrier gas at 66 ml per min. through a spiral copper column (4 mm internal diam.) packed with 30 to 60-mesh Linde mol. sieve activated *in vacuo* at 350°. At 20°, Ar, O and N are eluted after ≃6, 6·5 and 18 min., respectively.
CHEM. ABSTR.

3164. **Rapid method for separation and analysis of radioactive fission gases.** R. C. Koch and G. L. Grandy (Chem. Dept., Nucl. Sci. and Engng Corp., Pittsburgh 36, Pa., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 43–48.—Fission gases (^{133}Xe, ^{135}Xe, ^{85m}Kr and ^{87}Kr) in helium streams are separated and determined with a precision of 7%. A sample of the gas is collected on a charcoal sampling bed and then passed through a charcoal-filled gas-chromatography column in a helium carrier stream. The fractions are collected in traps at −80° and assayed radiometrically. Thus ^{138}Xe (17 min.) and ^{89}Kr are determined by measurement of their decay products ^{138}Cs (32 min.) and ^{89}Rb (17 min.), respectively. A correction for the effect of the parent isotope radioactivity is derived. J. P. STERN

3165. **Turbidimetric determination of potassium and ammonium in the presence of each other with sodium tetraphenylborate.** Z. Gregorowicz and F. Buhl (Inst. für anorg. Chem. der Schlesischen Tech. Hochsch., Gliwice, Poland). *Mikrochim. Acta*, 1961, (1), 129–133 (in German).—The influence of time and of HCl, acetic acid and NaOH on extinction values was examined and it was concluded that the test should be carried out in neutral or alkaline soln. The soln. containing K^+ and/or NH_4^+ is mixed with 2 ml of 0·1% gum acacia soln. and diluted to ≃20 ml. After addition of 2 ml of Na tetraphenylborate (**I**) soln. (2·5 g of **I** in 50 ml of water, shaken with Al_2O_3 and filtered), the soln. is diluted to 25 ml. After 25 min. the extinction is measured (Pulfrich photometer, filter S47); Beer's law is obeyed for 0·1 to 1 mg of K^+ per 25 ml and for 0·05 to 0·5 mg of NH_4^+ per 25 ml. To obtain K^+ alone, NH_4^+ are expelled by reaction with NaOH, and the soln. tested for K^+ as before.
P. D. PARR-RICHARD

3166. **A flotation method for studying the rubidium admixture effect on the density of potassium chloride crystals and the determination of rubidium content in potassium salts.** V. S. Rŷlov (Eng.-Phys. Inst., Acad. Sci. USSR, Leningrad). *Zhur. Anal. Khim.*, 1961, **16** (1), 14–21.—The flotation method was used to investigate the effect of impurities on the density of crystals, *e.g.*, RbCl admixtures in KCl. The distribution of an impurity between the molten salt and its crystal phase was studied. In KCl the distribution coeff. for RbCl depends on the Rb concn. and approximates to $\alpha = 1$ for low concn. of RbCl, and the density of KCl increases linearly with RbCl concn. This method is used for the determination of Rb in potassium chloride in the concn. range 5·0 to 0·001 atom %.
A. BURWOOD-SMITH

3167. **Flame-photometric determination of Clarke amounts of caesium in natural minerals rich in potassium.** E. A. Fabrikova (Inst. of Mineralogy, Geochem. and Crystallochem. of Rare Elements, Acad. Sci. USSR, Moscow). *Zhur. Anal. Khim.*, 1961, **16** (1), 22–24.—Caesium is co-pptd. with ammonium molybdosilicate from H_2SO_4 soln. *Procedure*—Dissolve 5 g of sample (containing ≃1 p.p.m. of Cs) in acid (40 to 50 ml of HF, 10 ml of H_2SO_4 and 1 ml of HNO_3) in a platinum dish. Add a further 5 ml of H_2SO_4 and evaporate to destroy HF. Cool, add 50 ml of H_2O, filter and wash the residue with warm water. Evaporate the filtrate and washings to 50 ml and acidify with 3 ml of H_2SO_4 (1·83). After careful mixing, add 15 ml of molybdosilicate reagent and 10 g of dry NH_4Cl to precipitate yellow ammonium molybdosilicate. Set aside for 30 min. then wash the ppt. 3 times with a soln. containing 500 ml of H_2O, 30 ml of H_2SO_4 (1·83) and 100 ml of NH_4Cl soln. Treat the ppt. with 30 ml of aq. NH_3 (1 : 1) and determine the Cs flame-photometrically (*Anal. Abstr.*, 1959, **6**, 4282). For the standard soln. take 50 ml of Cs_2SO_4 (1·5 μg of Cs per ml), acidify with 3 ml of H_2SO_4 and proceed as described above. Prepare the molybdosilicate reagent by dissolving 38 g of MoO_3 in 10% NaOH soln. (100 ml) and mix with a soln. containing 2·0 g of NaOH, 1·5 g of $Si(OH)_4$ and 50 ml of H_2O. Carefully mix the soln. obtained with 12 ml of H_2SO_4 (1·84) in 100 ml of H_2O. A clear bright-yellow soln. should be obtained.
A. BURWOOD-SMITH

3168. **Detection of copper by a new spot test.** P. Umapathy and N. Appala Raju (Chem. Dept., Sri Venkateswara Univ. Coll., Tirupati, India). *Curr. Sci.*, 1960, **29** (11), 428.—Resacetophenone phenylhydrazone (prep. described) gives a green ppt. in ammoniacal media with small amounts of Cu, and a brown one with larger amounts. The reaction does not take place in acid or neutral soln., and the concn. of NH_3, the amount of reagent and the order of addition affect the results. In media rendered alkaline with NaOH the ppt. is bright red in conc. soln. The following procedure is recommended. To a drop of test soln. on a spot-plate add 2 or 3 drops of 4N to 6N-aq. NH_3 and 2 drops of a 10% ethanolic soln. of reagent. With very small amounts of Cu a green colour develops slowly. The limits of detection and dilution are 0·5 μg and 1 in 10^5, respectively. R. E. E.

3169. **Respropiophenone oxime as a reagent for the determination of copper.** J. S. Dave and A. R. Patel (Fac. of Sci., M.S. Univ., Baroda, India). *Curr. Sci.*, 1960, **29** (12), 472.—Respropiophenone oxime (prep. described) reacts with Cu in acetic acid soln. to yield a brown ppt. of $Cu(C_9H_{10}O_2N)_2$. *Procedure*—Neutralise the soln. of $CuCl_2$ (10 ml) with 2N-NaOH, acidify with 2N-acetic acid, boil and add an excess of a 1% soln. of the reagent, with stirring. Heat the mixture to boiling, collect the ppt. in a sintered glass crucible, wash it with hot water containing acetic acid until the filtrate gives no colour with $FeCl_3$ soln. and dry it at 110° to 120°.
R. E. E.

3170. **Photometric determination of copper as a copper - azide complex.** K. Kapitańczyk, Z. Kurzawa and Z. Prymiński (Dept. of Gen. Chem., Politechnika, Poznań, Poland). *Chem. Anal., Warsaw*, 1961, **6** (1), 23–27.—A stable olive-green complex is formed by Cu^{2+} and azide. The extinction of the complex increases with rising concn. of azide, and maximum absorption is shown in the blue - violet spectral region at pH 5·0 to 7·0. Interference is caused by Co, Ni and Cr (which form coloured azides), Al, Bi, Sb, Sn, Ti, Mo and Zr (through hydrolysis), Ag, Pb and Hg (by pptn.), NH_4^+ (by reaction with Cu), and PO_4^{3-}, CN^-, oxalate, tartrate and citrate. Small amounts of Fe are masked with NaF. Concn. of 0·1 to 2 mg of Cu can be determined with a precision of ±2%;

this range can be extended to 5 mg of Cu, but with loss in precision. *Procedure*—To 50 ml of sample add 10 ml of satd. NaF soln. if milligram amounts of Fe are present, adjust the pH to 5 to 6, add 5 ml of 10% NaN_3 soln., make up to 100 ml and measure the extinction. P. BRYCH

3171. **Thermometric determination of copper.** J. Hrivňák and J. Kováč (Res. Inst. Agrochem. Technology, Bratislava, Czechoslovakia). *Chem. Prümysl*, 1961, **11** (1), 27–28.—*Procedure*—Into a thermally insulated vessel with two apertures (for thermometer and for adding iron) measure a known vol. of the sample, as $CuCl_2$. Read the temp. and add reduced iron (4 g). Shake the mixture and register the increase of the temp. during the reaction $CuCl_2 + Fe = FeCl_2 + Cu - 37{,}300$ cal. per mole. Compare with a calibration curve. The method is suitable for the determination of the copper content of $CuCl_2$ in the presence of NaCl during the preparation of fungicides. The relative error is $\gtrsim \pm 1{\cdot}8\%$, and the time required for a determination 5 min. J. ZÝKA

3172. **Rapid determination of copper in alkaline copper and brass baths.** I. Dubský. *Metalloberfläche*, 1961, **15** (1), 13–15.—Hydrogen peroxide is used to oxidise free and combined cyanide, and to demask cations. Copper (or, in brass baths, Cu plus Zn) is then determined in soln. buffered with pyridine acetate, in the presence of glycine, by titration with EDTA (disodium salt), with xylenol orange or methylthymol blue as indicator. Zinc is determined separately in brass baths, in the presence of acetone and ammonia buffer, by a similar titration, with Eriochrome black T as indicator. G. P. MITCHELL

3173. **Methods for the analysis of copper alloys. Parts 1–5. Copper, lead, iron, aluminium and nickel.** British Standards Institution (2 Park Street, London, W.1). B.S. 1748: Parts 1–5: 1961. 21 pp.—The methods described are applicable to copper alloys containing 1 to 14% of Sn, up to 2% of Ni and small amounts of Al.

3174. **Flame-photometric determination of copper in mineral mixes. Ion-exchange technique for sample preparation.** W. G. Schrenk, K. Graber and R. Johnson (Agric. Exp. Sta., State Univ., Manhattan, Kans., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 106–108.—Copper is isolated from interfering ions by adsorption from 5M-HCl on Dowex I-X8, washing the column with 4M-HCl and then eluting (0·4 ml per min.) with 1·2M-HCl. The eluate is used directly for flame photometry, and Cu (0·03 and 0·06%) is determined at 324·7 mμ, by use of a calibration curve, with an accuracy within ±5% and ±2·8%, respectively. J. P. STERN

3175. **Determination of copper and manganese in ores by neutron-activation analysis.** E. M. Lobanov, O. M. Romanov, M. M. Romanov and A. A. Khaldarov (Inst. of Nuclear Phys., Acad. Sci. UzbekSSR, Tashkent). *Zhur. Anal. Khim.*, 1961, **16** (1), 25–28.—A γ-ray scintillation spectrometer is used for recording the γ-ray radiation of activated rock samples at a neutron flux of about 10^7 per sq. cm per sec. The determination of 0·03 to 0·9% of Cu as ^{64}Cu and 0·028 to 0·3% of Mn as ^{56}Mn is possible with an error of ±8%. A. BURWOOD-SMITH

3176. **Colorimetric determination of some metals with Reinecke salt. II. Determination of silver.** J. Weyers (Dept. of Inorg. and Anal. Chem., Med. Acad., Kraków, Poland). *Chem. Anal., Warsaw*, 1960, **5** (6), 979–983.—*Procedure*—Dissolve the sample in conc. H_2SO_4 and H_2O_2 by heating, and to the boiling soln. add satd. Reinecke salt soln. Filter off the Ag reineckate on a sintered glass filter, wash it with acetone and dissolve it in pyridine. Measure the extinction of the pyridine soln. with a green filter and a 2-cm cell. Compare with a calibration curve. Beer's law is obeyed over the range 3 to 15 μg of Ag per ml. The error was $\gtrsim 3{\cdot}3\%$. B. K.

3177. **Automatic direct-reading X-ray spectrometry. Applications to determinations of silver.** J. E. Moore, G. P. Happ and D. W. Stewart (Res. Lab., Eastman Kodak Co., Rochester, N.Y., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 61–64.—The print-out system described may be added to existing manual spectrometers. Through the use of commercially available circuits, it provides semi-automatic compensation for background and, over the range of linear calibration, printed analytical results in direct weight units of the element being determined. Automatic sample handling is provided by mechanical changes in the sample compartment. E. G. CUMMINS

3178. **Rapid method for determination of small amounts of gold in the presence of silver, copper and nickel.** Z. Szponar (Dept. of Tech. Analysis, Polytech., Gdańsk, Poland). *Chem. Anal., Warsaw*, 1960, **5** (6), 973–977.—*Procedure*—The sample is dissolved in aqua regia and the soln. is partially evaporated and diluted with H_2O. The ppt. of AgCl is filtered off and the $AuCl_4^-$ are separated by paper electrophoresis for 45 min. at 11·5 V per cm in 0·1N-KCl. The spot of $AuCl_4^-$ is located on a marker strip with 0·05% benzidine soln. in 10% acetic acid. Spots are cut out from other strips and the gold salts are dissolved in H_2O. To these soln. 0·01% magenta soln. is added, and after 40 min. the extinction is measured in a Pulfrich photometer with a S50 filter and referred to a calibration curve; 1 to 250 μg of Au can be determined. The coeff. of variation is 0·31% (7 determinations). B. K.

3179. **Semi-quantitative determination of beryllium in rocks and soils by paper chromatography.** H. Agrinier (Centre d'Études Nucléaires, Fontenay-aux-Roses). *Chim. Anal.*, 1960, **42** (12), 600–602.—The sample (1 g) (75 μ) is digested with HF (4 ml) and H_2SO_4 (2 ml) for 30 min. and evaporated to dryness; the residue is treated for 15 min. with HNO_3 (sp. gr. 1·33) (4 ml) and H_2O_2 (110 vol.) (0·5 ml) before being again taken to dryness, re-attacked with HNO_3 (4 ml) and re-evaporated to dryness; the residue is treated with HNO_3 (2·5 ml). The soln. is filtered and 0·025 ml is applied to a C.R.L. pattern paper. The chromatograms are developed for 1 hr. in a solvent mixture of acetone (65 ml) and HNO_3 (35 ml), dried for 10 min. at 35°, exposed to NH_3 vapour for 2 min. and sprayed with 0·2% quinalizarin in acetone - pyridine (9:1). The intensity of colour in the Be band is compared with standards. Results on rocks (11 samples) agree with spectrographic figures within ±10% (5 to 100 p.p.m. of Be). T. R. ANDREW

3180. **Determination of hydrogen in Magnox A12.** R. Berry, J. A. J. Walker and A. E. Clarke [U.K.A.E.A. (Reactor Technology Branch), Culcheth]. U.K.A.E.A. Report DEG 107(C), 1961. 19 pp.—Experiments to decide between three methods are described. They are (*i*) vacuum

extraction at 550°, (*ii*) vacuum sublimation at 750°, and (*iii*) vacuum fusion in a tin bath at 400°. Of these, (*ii*) is preferred for routine analyses and gives the most consistent results; full experimental details of the procedure are appended. In 12 analyses involving from 1·18 to 1·56 g of Magnox A12, the average content of H was 0·099 ml per g and the standard deviation was 0·020 ml per g.

G. J. HUNTER

3181. **Simultaneous complexometric determination of calcium and magnesium.** B. Bieber and Z. Večeřa (State Res. Inst. of Raw Materials and Technol., Brno, Czechoslovakia). *Chem. Anal., Warsaw*, 1960, **6** (1), 17–22.—*Procedure*—To about 100 ml of sample add thymolphthalein soln., make alkaline with, and add a 20-ml excess of, 20% KOH soln., then add a few crystals of KCN and 1 ml of calcein soln. (0·1%) and titrate with EDTA (disodium salt) soln. until the green fluorescence becomes purple-red (reflected light). The titre is equiv. to the Ca content. To the soln. made yellow with HCl add a few crystals of hydroxyammonium chloride and aq. NH_3 till the soln. is green, and titrate with EDTA to Eriochrome black T. The pptn. of Mg with KOH when the Mg to Ca ratio is >2:1 is prevented by the addition of tartaric acid. Determinations of Ca and Mg in limestone, basic and acid slags, dolomite and magnesite differed by ≯0·30% from results obtained gravimetrically.

P. BRYCH

3182. **Separation of calcium and magnesium followed by complexometric determination.** B. Bieber and Z. Večeřa (State Res. Inst. of Raw Materials and Technol., Brno, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1961, **26** (1), 59–66 (in German).—Calcium was separated from Mg as $CaSO_3$ (**I**), $CaWO_4$ (**II**) and Ca oxalate (**III**) by modifications of standard procedures. Magnesium was determined in the filtrates by titration with EDTA (disodium salt), with Eriochrome black T as indicator. **I** was dissolved in acid, treated with ammoniacal buffer and cyanide, and the Ca was titrated in the same way; **II** and **III** were heated with an excess of ammoniacal EDTA soln. and, after adding hydroxyammonium chloride, the excess was back-titrated with standard Zn acetate soln., with the same indicator. Results obtained on slag, dolomite and magnesite agreed well with those by standard procedures.

J. W. PRICE

3183. **Complexometric titrations (chelatometry). XLIX. Determination of calcium and magnesium in the presence of large amounts of manganese.** P. Povondra and R. Přibil (Czech. Acad. Sci., Prague). *Coll. Czech. Chem. Commun.*, 1961, **26** (1), 311–314 (in German).—Manganese in the form of the cyanide complex $[Mn(CN)_6]^{3-}$ does not react with EDTA and in this form may be present in amounts of 80 to 100 mg without interfering with the titration of up to 46 mg of Ca and up to 25 mg of Mg. *Procedure*—For each 30 mg of Mn expected take 10 ml of a 20% triethanolamine soln. (**I**), and dilute with 60 ml of H_2O. For each 10 ml of **I** add 10% NaOH soln. (5 ml) to adjust the pH to 12·5. Add the soln. to be analysed slowly, with rapid stirring, and then add KCN (3 g). Add dil. acetic acid until the colour changes from green to brownish yellow (pH 11) and then add 15 ml of aq. NH_3 - NH_4Cl buffer soln. and 20 ml of conc. aq. NH_3. To the clear soln. add a known excess of 0·05M-EDTA (disodium salt) and titrate the excess with 0·05M-Ca^{2+} to a deep-blue colour with thymolphthalein complexan (Thymolphthalexone) as indicator.

J. W. PRICE

3184. **Limits of interference by iron, manganese, aluminium and phosphate in the EDTA determination of calcium in the presence of magnesium using Cal-red as indicator.** P. Moss (Imperial Coll. of Tropical Agric., St. Augustine, Trinidad, B.W.I.). *J. Sci. Food Agric.*, 1961, **12** (1), 30–33.—Under the prescribed conditions for the determination of Ca (0·1 milli-equiv.) in plant and soil extracts, up to 2 mg of Fe, 1 mg of Mn, 3 mg of Al or 6 mg of phosphate can be tolerated in 50 ml of soln. *Procedure*—To a mixture containing 0·02N-$CaCl_2$ (5 ml) and 0·02N-$MgSO_4$ (5 ml) add 8N-NaOH (4 ml), 1·5M-KCN (2 ml), 5% hydroxyammonium chloride soln. (2 ml) and 30% triethanolamine soln. (1 ml). Make up to 50 ml with water, set aside for 1 hr., add 45 to 50 mg of indicator [Cal-red - K_2SO_4 (1:100)] and titrate with 0·02N-EDTA (disodium salt) to a permanent blue end-point.

M. A. ELLIS

3185. **Thermometric precipitation titration of calcium in the presence of magnesium. Kinetic masking and application to limestone analysis.** J. Jordan and E. J. Billingham, jun. (Dept. of Chem., State Univ., University Park, Pa., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 120–123.—Thermometric determination of Ca in presence of Mg in borate buffer soln. of pH 8 is possible by rapid titration with 0·2M-ammonium oxalate because of the slow pptn. of the Mg due to the formation of an intermediate. By the procedure given, 5×10^{-3} to 10^{-1} mole of Ca may be determined with an accuracy within ±1% in the presence of Mg (<0·1 mole), Al (10^{-3} mole) and Fe (2×10^{-3} mole).

J. P. STERN

3186. **Diphenylcarbazone as an internal indicator in volumetric analysis. Determination of calcium.** G. S. Deshmukh, M. G. Bapat and A. L. J. Rao (Benares Hindu Univ., Varanasi, India). *Z. anal. Chem.*, 1961, **179** (1), 18–21 (in English).—*Procedure*—To a soln. containing 20 to 100 mg of Ca add excess of 0·1N-Na oxalate, ethanol (2 ml) and 5 drops of diphenylcarbazone (satd. soln. in ethanol). Titrate the unused Na oxalate soln. with $Pb(NO_3)_2$ soln. to a cherry-red colour. Barium and Mg do not interfere.

J. W. PRICE

3187. **The use of strontium as an internal standard and buffer in the flame-spectrometric determination of calcium in the presence of various interfering elements.** J. Fischer and R. Kropp (Inst. Inorg. Chem., Goethe Univ., Frankfurt, Germany). *Z. anal. Chem.*, 1961, **179** (1), 1–12 (in German).—The method described is applicable to the rapid determination of Ca without separation in such material as blast-furnace slags, cement and fertilisers. *Procedure*—Dissolve 0·1 g of sample by heating with 5 ml of HCl and 10 ml of H_2O. Siliceous materials are first treated with HF, F^- are removed by evaporation with $HClO_4$ (1 to 1·5 ml) and the residue is dissolved in 10 drops of HCl (1:1) and 10 to 20 ml of H_2O. Dilute the soln. to 500 ml (for concn. of Ca <10% dilute to 100 ml) and to an aliquot containing 0·1 to 0·5 mg of Ca add 5·0 ml of a strontium soln. (≡1 g of Sr per litre), 10 drops of HCl (1:1) and H_2O to 50 ml (pH 1·2). Measure the Ca emission of the soln. at 554 mμ (in the presence of excess of Fe measure at

422·7 mμ) and the Sr emission at 460·7 mμ. Prepare calibration curves for 0 to 10 mg of Ca per litre plus 100 mg of Sr per litre and for 20 to 100 mg of Sr per litre. Correct the reading for Ca by dividing by the apparent content of Sr and multiplying by 100, and obtain the content of Ca from the calibration curve and the corrected reading. The interference of Al, SiO_2, H_2SO_4, H_3PO_4, Fe and U is thus allowed for. J. W. PRICE

3188. **Quantitative radiochemical analysis by ion exchange. Calcium, strontium and barium.** L. Wish (U.S. Naval Radiological Defense Lab., San Francisco, Calif., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 53–55.—The separation of Ce, Ca, Sr and Ba from the rare-earth metals and one another is achieved at room temp. on 8% cross-linked Dowex 50 by elution with ammonium 2-methyl-lactate soln. of pH 4·13, 4·15, 5·0 and 6·2, respectively, by the technique of Baerg and Bartholomew (*Canad. J. Chem.*, 1957, **35**, 980). The method is applied to the analysis of fission products, after removal of Ru by volatilisation of RuO_4 and of Zr, Nb, Mo, U, Np, Te, Pu, Ag, Cd and Rh as their chlorides on Dowex 2. The average recovery is 99·8% and the coeff. of variation is 0·8%. J. P. STERN

3189. **Determination of gypsum and its dehydration products in their mixtures.** H. G. McAdie and R. A. Kuntze (Ontario Res. Foundation, Toronto, Canada). *Mater. Res. & Stand.*, 1961, **1** (1), 27–29.—Procedures are given for determining, per g of dried plaster, (*i*) the wt. of combined H_2O, and (*ii*) the gain in wt. after hydration of the sol. anhydrite (**I**) to the hemihydrate (**II**) with 80% (v/v) aq. ethanol. By applying these two values to the triangular diagram for the $CaSO_4$ - H_2O system, the proportions of **I**, **II** and dihydrate (**III**) can be determined directly or from equations. For impure samples it is necessary to determine (as described) the $CaSO_4$ and H_2O present as **I**, **II** and **III**, but impurities (*e.g.*, accelerators, retarders and surfactants) that change in wt. at temp. $<225°$ interfere. The results are satisfactory and the separate hydration and dehydration procedures require only $\simeq 12$ hr. instead of the three days required for equilibration with water vapour.
W. J. BAKER

3190. **Separation of strontium from calcium by selective precipitation of strontium sulphate.** L. Berák and J. Münich (Czech. Acad. Sci., Prague). *Coll. Czech. Chem. Commun.*, 1961, **26** (1), 276–281 (in German).—The addition of a soln. of $MgSO_4$ to mixtures of the Ca and Sr complexes with EDTA liberates the Sr from its complex, with pptn. of $SrSO_4$. The completeness of pptn. of Sr and the co-pptn. of Ca were investigated by means of the radio-isotopes ^{45}Ca, ^{89}Sr and ^{90}Sr. At a Ca to Sr ratio of 1:1 a single pptn. gives good results at pH 10 but, in the presence of an excess of Ca, increasing amounts of Ca are co-pptd., and better results are obtained at pH 8·5, only 2% of the Ca being pptd. at a ratio of 9:1. When the ratio is $>1:1$ add 1 milli-equiv. of EDTA (disodium salt) to the soln. containing 1 milli-equiv. of $MgSO_4$ and dilute to 100 ml. Set aside for 2 to 4 hr., filter off the $SrSO_4$ and titrate the Mg in the filtrate with EDTA, with Eriochrome black T as indicator. With an excess of Ca add sufficient buffer soln. to give a red colour with phenolphthalein before pptg. the $SrSO_4$. The method can be used for the separation of Ba from Ca, but not of Ba from Sr. J. W. PRICE

3191. **Simplified determination of strontium-90. Preferential extraction of yttrium with tributyl phosphate.** R. J. Velten and A. S. Goldin (Robert A. Taft Sanitary Engng Center, Cincinnati 26, Ohio, U.S.A.). *Anal. Chem.*, 1961, **33** (1), 128–131.—The preferential extraction of Y from 14N-HNO_3 soln. by tributyl phosphate (**I**) permits the more rapid determination of ^{90}Sr by γ-spectroscopy, especially in samples in which ^{90}Sr has been concentrated, *e.g.*, biological materials in which metabolic rejection of lanthanides, Zr, Nb and Ru has occurred, water samples after sedimentation, and samples older than 400 days in which interfering elements (except ^{91}Y and ^{161}Pm) have decayed. Results on milk containing 2·9 to 18·6 μμC per litre were of the same order as those obtained by the standard method. *Procedure*—To the sample in 10% HCl or HNO_3 add Ca (0·5 to 1 g) and excess of oxalic acid. Adjust the pH of the almost boiling soln. to 2·5 to 3, and centrifuge and wash the ppt. Dissolve the ppt. in conc. HNO_3 (20 ml), evaporate to 15 ml, and extract with **I** (3 × 15 ml). Strip Y from the organic phase with 0·1N-HNO_3 and determine it radiometrically. J. P. STERN

3192. **Application of tributyl phosphate extraction to the determination of strontium-90.** A. S. Goldin and R. J. Velten (Robert A. Taft Sanitary Engng Center, Cincinnati 26, Ohio, U.S.A.). *Anal. Chem.*, 1961, **33** (1), 149.—The quant. extraction of Y from 12N-HNO_3 soln. by tributyl phosphate and its subsequent stripping from the organic phase by 0·1N-HNO_3 is reported; ^{90}Y can be separated from La and Ce by this method. The procedure may be utilised for the determination of ^{90}Sr.
J. P. STERN

3193. **Separation of zinc from aluminium and titanium and its determination.** L. Giuffrè and F. M. Capizzi (Polytech., Milan, Italy). *Ann. Chim., Roma*, 1960, **50** (11), 1552–1557.—The soln. containing Zn, Al and Ti, acidified with H_2SO_4, is treated with H_2O_2 soln. (3%) and aq. 0·1M-EDTA (disodium salt). To separate the Zn and Al from Ti, the pH is adjusted to <2, or to separate Zn from Al and Ti, to 3·5. The soln. is then passed through a cation-exchange column. In the first case the Ti complex will pass through, the Zn^{2+} and Al^{3+} remaining on the column; in the second case the Ti and Al complexes will pass through, leaving the Zn^{2+} on the column. The Zn is finally eluted with 4M-HCl, the eluate is treated with H_2SO_4 - HNO_3 to destroy residual EDTA, and the Zn is determined complexometrically or spectrophotometrically. L. A. O'NEILL

3194. **Chromatographic separation of zinc and cadmium on ion exchangers.** M. Marhoul (Inst. Nucl. Res., Acad. Sci., Prague). *Chem. Prûmysl*, 1961, **11** (2), 102–104.—Strongly acid polystyrene cation exchangers with varying proportions of divinylbenzene (2 to 30%) were prepared and their use for the separation of Cd^{2+} and Zn^{2+} was studied. For quant. separations the best were those containing from 6 to 8% of divinylbenzene, with 0·1M-citric acid (pH 4·4 to 4·6) as eluent (1 ml per min.).
J. ZÝKA

3195. **Micro-determination of mercury with thioacetamide.** E. Bovalini and M. Piazzi (Lab. Chim. Accad. Navale, Livorno, Italy). *Ann. Chim., Roma*, 1960, **50** (11), 1449–1452.—The Hg^{2+} are complexed with EDTA and pptd. as HgS with standard thioacetamide solution (0·01 to 0·001M)

at pH 8·5 to 9·0, the end-point being determined heterometrically. Zinc, Cd and Co do not form sulphides under the conditions employed; 100 μg of Hg may be determined. L. A. O'NEILL

3196. **Hydrazine sulphate as a reagent in volumetric analysis (hydrazinometry). VII. Determination of mercury(II).** L. Vulterin (Inst. Chem. Ind., Tech. Hochsch., Prague, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1961, **26** (1), 317–320 (in German).—*Procedure*—To the soln. containing 10 to 300 mg of Hg^{II} add solid KI or KBr (0·5 to 1 g) until the ppt. re-dissolves, and then add KOH soln. to give a concn. of 23 to 33% for the iodide or 0·75 to 5% for the bromide. Titrate to a potentiometric end-point with 0·1N-hydrazine sulphate. Saturated soln. of NO_3^-, SO_4^{2-}, CO_3^{2-}, F^-, Cl^- Br^- as Na or K salts do not interfere, but ammonium salts must be removed. Cyanides interfere. The average deviation is 0·35%. J. W. PRICE

3197. **Investigation of the benzoin method for the fluorimetric determination of boron.** G. Elliott and J. A. Radley (Radley Industrial Res. and Development Lab., 220 Elgar Road, Reading, Berks., England). *Analyst*, 1961, **86**, 62–69.—The fluorescence of the boron - benzoin complex used by White and Neustadt (*Ind. Eng. Chem., Anal. Ed.*, 1943, **15**, 599), White *et al.* (*Anal. Chem.*, 1947, **19**, 802) and White and Hoffman (*Ibid.*, 1957, **29**, 1105) for determining amounts of boron from 1 μg upwards is investigated. The development and stability of the boron - benzoin complex in ethanol and other solvents is studied with particular attention to the de-activating effect of oxygen. With formamide and its *N*-methyl derivatives as solvents, the fluorescence is stronger than that in ethanol. A glycine buffer soln. (pH 12·8) produces the correct conditions for developing fluorescence with ethanol as solvent, but not with the formamide series of solvents. Isopropylamine and isobutylamine are effective bases in both the ethanol and the formamide series. With solvents of a given chemical type (*e.g.*, the formamides) there may be increase of fluorescence intensity with dielectric constant, but this is not true for the alcohols. The inhibiting effect of oxygen on the development of fluorescence is much less in formamide than in ethanol. The relationship between the fluorescence intensity and amount of boron is linear in the range studied (0·05 to 0·5 μg). A. O. JONES

3198. **Coulometric determination of boric oxide in heavy water.** A. Parker and E. A. Terry (U.K.A.E.A., Res. Group, C.37 Royal Arsenal, Woolwich, S.E.18). Analytical Method AERE-AM 72, 1961. 6 pp.—A basal soln. (30 ml) containing KCl (7·4% w/v) and mannitol (10% w/v) is placed in a special cell (illustrated) and current is passed until the pH is between 8·5 and 9. The sample, containing 10 to 50 mg of B_2O_3, is then added and the number of coulombs required to restore the pH to the value reached in the first step is determined, whence the weight of B_2O_3 may be derived. The coeff. of variation is 0·3% for a sample containing 25 mg of B_2O_3. The method is not applicable to soln. containing sources of acidity other than $^2H_3BO_3$, or ions more easily reduced than $^2H^+$ in the pH range 3 to 9. G. J. HUNTER

3199. **Colorimetric determination of aluminium 8-quinolinolate in chloroform.** R. H. Linnell and F. H. Raab (Dept. of Chem., Univ., Burlington, Vt., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 154–155.—Measurable decomposition of the Al - 8-hydroxyquinoline chelate occurs even in the dark within 1 hr. Decomposition is faster in O and light, and faster in commercial $CHCl_3$ than in pure $CHCl_3$; it is suggested that decomposition products of $CHCl_3$, *e.g.*, $COCl_2$, may be responsible for the decomposition of the chelate. J. P. STERN

3200. **Determination of traces of copper in pure aluminium and indium by means of nickel diethyl dithiophosphate.** A. I. Busev and N. P. Borzenkova (M.V. Lomonosov Moscow State Univ.). *Zavod. Lab.*, 1961, **27** (1), 13–15.—*Procedure with metallic aluminium*—The sample (1 g) is dissolved by heating in 10N-H_2SO_4 and the soln. is diluted so that the acid concn. is N and the vol. is 100 ml. It is then extracted with 5 to 10 ml of CCl_4 in the presence of 1 to 5 ml of aq. 0·0005M-Ni diethyl dithiophosphate. Two further extractions are made and the combined extracts are diluted to between 10 and 25 ml. The extinction is measured at 420 mμ. *Procedure with metallic indium*—The sample (1 g) is dissolved in 20 ml of H_2SO_4 (1 : 1) and the soln., after dilution to an acid concn. of N, is extracted with 5 ml of CCl_4 and 1 ml of the reagent soln. A special cell is described for the determination of very low concn. (< 0·2 μg of Cu per ml). G. S. SMITH

3201. **Analysis of aluminium sulphate.** J. Navarro Sagrista. *Inf. Quim. Anal.*, 1961, **15** (1), 14–20.—Procedures are given for the following determinations—material insoluble in water, combined Al_2O_3 (by titration with NaOH), total Al_2O_3 (gravimetric and complexometric), total H_2SO_4, free acid and Al_2O_3, free occluded acid, total Fe (complexometric, permanganate and colorimetric) and Fe^{III}. G. H. FOXLEY

3202. ***NN'*-Di-(2-hydroxy-5-sulphophenyl)-*C*-cyanoformazan as a reagent for the photometric determination of gallium.** M. I. Ermakova, N. L. Vasil'eva and I. Ya. Postovskii (Inst. of Chem., Ural Branch of Acad. Sci. USSR, Sverdlovsk). *Zhur. Anal. Khim.*, 1961, **16** (1), 8–13.—A method for the synthesis of *NN'*-di-(2-hydroxy-5-sulphophenyl)-*C*-cyanoformazan (**I**) and its use for the photometric determination of Ga are described. At pH 3 the absorption band of the complex with Ga (634 mμ) is distinct from that of **I** (470 mμ); the molar extinction coeff. at pH 3 and 634 mμ is 25,000. Soln. of the complex obey Beer's law for concn. of 0·05 to 2·5 μg of Ga per ml. **I** is sol. in H_2O and ethanol, but not in ether, benzene, acetone, CCl_4 or $CHCl_3$. With this reagent the determination of Ga in aluminium and zinc is possible when the ratios of Ga to Al and of Ga to Zn are 1 : 50,000 and 1 : 5000, respectively. A. BURWOOD-SMITH

3203. **Polarographic determination of indium in gallium metal.** S. I. Sinyakova, N. A. Rudnev, Yü-Ch'ih Shen and R. Dzhumaev (V.I. Vernadskii Inst. of Geochem. and Anal. Chem., Acad. Sci. USSR, Moscow). *Zhur. Anal. Khim.*, 1961, **16** (1), 32–35.—*Procedure*—Dissolve 2 to 5 g of gallium by boiling in aqua regia (25 to 30 ml) added in 10-ml portions. Evaporate the soln. to ≃5 ml, and dilute with 50 ml of H_2O. Add 10 to 25 g of tartaric acid and adjust the pH to 7 to 8 with aq. NH_3. Add 0·1M-$CoCl_2$ (1·7 ml per g of Ga) and co-precipitate In with CoS by adding 0·5N-Na_2S (10 to 25 ml).

Set aside for 30 min. to coagulate the ppt., centrifuge and wash the ppt. 3 or 4 times with 1% NH_4NO_3 soln. Dissolve the ppt. in 10 to 15 ml of conc. HNO_3, evaporate to dryness, and dissolve the residue in 2 to 3 ml of conc. HCl. Add 10 ml of H_2O and 2 g of sulphosalicylic acid and adjust the pH to 4·8 to 5·2. Extract the In twice with 3 to 5 ml of 0·05% dithizone soln. in CCl_4, wash the extract twice with water, then add 3 to 5 ml of 2M-HCl to separate the indium dithizonate and reject the CCl_4. Evaporate the HCl soln. to dryness, dissolve the residue in a few drops of conc. $HClO_4$ and re-evaporate to destroy organic substances. Dissolve the residue in 1 ml of 0·2M-HCl, de-aerate with N or H for 10 min. and record the polarogram from 0·2 to 0·8 V. This method and also an oscillographic method permit determinations of as little as 10^{-5} to 10^{-6}% of In with an accuracy of ±15%. A. BURWOOD-SMITH

3204. **Polarographic determination of indium in the presence of some organic acids.** E. N. Vinogradova and Ching-Ju An (Univ., Moscow, USSR). *Vestn. Moskov. Univ., Ser. Khim.*, 1960, No. 6, 52–57.—To a 1% soln. of In in conc. $HClO_4$ add succinic acid (**I**) (final concn. of **I** = 0·05M to 0·3M, pH = 2·6 to 3·5). Record the polarogram *vs.* the S.C.E. at 25°. Within the range 2×10^{-5} to 2×10^{-4} moles of In the diffusion current is proportional to concn. There is no interference from Zn, even in a 10,000-fold excess; if Pb (>10-fold excess) is present, precipitate it as $PbSO_4$. Remove Sn by successive evaporations of the sample with HCl - HBr, HCl, and HNO_3. Dissolve the residue in HCl, then add **I** (concn. and pH as above), and record the polarogram. If Sb is present, dissolve the alloy (10 to 60 mg) in conc. H_2SO_4 (15 ml) and heat on a sand bath. Cool, add citric acid (**II**) (final concn. of **II** = 0·01M to 0·2M) and HCl (1:1) to pH 0·3 to 1·0. Record the polarogram; the concn. of In is proportional to the diffusion current. If Sb is present in more than a 20-fold excess, it must be removed, *e.g.*, by concentration on zinc amalgam. J. G. GORODI

3205. **Colorimetric determination of traces of indium in silicate minerals with dithizone and 5,7-dibromo-8-hydroxyquinoline.** J. Minczewski, U. Stolarczyk and Z. Marczenko (Dept. of Anal. Chem., Politechnika, Warsaw). *Chem. Anal., Warsaw*, 1961, **6** (1), 51–62.—The method of Luke and Campbell (*Anal. Abstr.*, 1957, **4**, 66) has been modified to prevent interference by the large excess of Al and Fe present in these ores, and the intensity of the developed colour is improved by substituting 5,7-dibromo-8-hydroxyquinoline (**I**) for 8-hydroxyquinoline. *Procedure*—The ore is dissolved in $HClO_4$ and HF, sufficient 10% sulphosalicylic acid (neutralised to pH 9 with aq. NH_3) is added for masking (a 3-fold excess for Al and a 10-fold excess for Fe), followed by 1 ml of 10% hydroxyammonium chloride soln. and aq. NH_3 to pH 9. Cyanide reagent (253 ml of 2N-aq. NH_3, 377 ml of 2N-HNO_3, 200 ml of 10% KCN soln., and water to 1 litre) (10 ml) is added as masking agent for other metals. Indium is extracted with dithizone soln. in $CHCl_3$ (0·01%) (4 × 5 ml) and re-extracted with 20 ml of 1% HNO_3. The extract is neutralised to methyl orange with aq. NH_3, and buffered with 25 ml of phthalate buffer (pH 3·5), and In is extracted with 0·1% **I** soln. in $CHCl_3$ (2 × 10 ml). The extracts are made up to 25 ml with extracting soln. and the extinction is measured at 450 mμ. An inherent systematic error of about 5% is due to losses during extraction, and the relative error is ±3%. The limit of determination of In is 0·001%. P. BRYCH

3206. **Qualitative test for thallium with iodide ions in the presence of masking agents.** J. Ramírez-Muñoz and L. Capacho-Delgado (Univ. Ind. de Santander, Bucaramanga, Colombia). *Rev. Univ. Ind. Santander, Bucaramanga, Colombia*, 1960, **2** (2–3), 105–128.—The sensitivity of the reaction with I^- for the detection of Tl has been investigated in the presence of Ag^+ and Pb^{2+} with tartrate and CN^- as masking agents. *Procedure*—To a drop of the neutral or slightly acid test soln. add a drop of KI soln. (15%) followed by a drop of Na K tartrate soln. (20%) and a drop of KCN soln. (10%), mix well, and examine the mixture against a black background with lateral illumination. A yellow precipitate indicates the presence of Tl. The limit of detection is $10^{-4.6}$ to $10^{-4.3}$ g-ion of Tl per litre. G. H. FOXLEY

3207. **Basic problems of complexometry. IV. Determination of thallium.** R. Přibil, V. Veselý and K. Kratochvíl (Inst. of Geochem. and Inorg. Chem., Czech. Acad. Sci., Prague). *Talanta*, 1961, **8** (1), 52–54.—The titration of Tl^I and Tl^{III} in alkaline medium is not reliable with Eriochrome black T as indicator, as the colour change is not sufficiently sharp; Tl^{III} can be reliably determined in the presence of Tl^I by direct titration with EDTA, with xylenol orange as indicator, in a weak acid medium. A second aliquot of the soln. can be used for the determination of Tl^I by potentiometric titration with $KBrO_3$. Another procedure depends on evaporation of the sample with aqua regia followed by the complexometric determination of total Tl. C. B. BAINES

3208. **Determination of small amounts of thallium in minerals.** N. A. Rudnev, G. I. Malofeeva and V. S. Rasskazova (V.I. Vernadskiĭ Inst. of Geochem. and Anal. Chem., Acad. Sci., USSR). *Zavod. Lab.*, 1961, **27** (1), 20–21.—The use of Bi_2S_3 together with K tetraphenylborate as a collector for microgram amounts of Tl in 0·075N to 4·8N-H_2SO_4 is recommended. The ppt. is free from Ga and Sb. G. S. SMITH

3209. **Photometric determination of scandium with 4-(2-pyridylazo)resorcinol.** A. I. Busev and Fan Chang (Univ., Moscow, USSR). *Vestn. Moskov. Univ., Ser. Khim.*, 1960, No. 6, 46–51.—The method is based on the formation of a red sol. complex from equimolar proportions of Sc^{3+} and 4-(2-pyridylazo)resorcinol (**I**). Dissolve the sample in HCl, and neutralise. To 12 ml of the soln. add 2·5 ml of ammonium acetate soln. (20%) as buffer and 2·0 ml of **I** soln. (0·05%) and make up to 25 ml. The final concn. of Sc should be 0·05 to 2·0 μg per ml and the pH 6·0 to 7·3. Measure the extinction at 515 mμ. Interfering elements are Zn, Co, Ni, Cd, Cu, Tl, In, Ga, Zr, Th, U and Fe. Calibrate the photometer with pure Sc_2O_3. J. G. GORODI

3210. **Colorimetric determination of cerium(IV) with 2-thio-5-vanillylidenehydantoin.** F. Pino-Pérez, F. Burriel-Martí and J. Valero (Dept. de Quím. Anal. del C.S.I.C. Madrid y La Laguna). *Inf. Quim. Anal.*, 1961, **15** (1), 1–13.—In acid soln. Ce^{4+} react with 2-thio-5-vanillylidenehydantoin (**I**) to form a complex having an absorption maximum at 520 to 530 mμ and obeying Beer's law

over the range 0 to 10 mg of Ce per 50 ml. *Procedure*—The Ce is pptd. as $CeO_2.xH_2O$, which is separated by centrifuging and dissolved in N-H_2SO_4 to give a soln. containing ≃1 mg of Ce per ml. A suitable volume of this soln. containing up to 7 mg of Ce is added to the reagent soln. (25 ml of a soln. of 0·025 g of **I** in 100 ml of methanol), and diluted to 50 ml. The extinction is measured at 520 mμ. Oxidising agents, H_3PO_4 and alkali-metal salts interfere. The precision of the method is ≃5%.
G. H. FOXLEY

3211. **The polarographic behaviour of cerium(IV) and the cerium(IV) - cerium(III) system in sulphuric media. A direct determination of cerium(IV).** P. G. Desideri (Inst. Anal. Chem., Univ. of Florence, Italy). *J. Electroanal. Chem.*, 1961, **2** (1), 39–45.—The development of the bubbling platinum electrode (Cozzi and Desideri, *Anal. Abstr.*, 1961, **8**, 880) has made possible the observation of $E_{\frac{1}{2}}$ values for Ce^{4+} and their variation with conditions. The reduction is semi-kinetic in character in M-H_2SO_4 soln. and a stabilising time (7 min.) is necessary before recording the polarogram. The temp. coeff. of the diffusion current is +4·3% per degree. The addition of a catalyst (ICl, 10^{-6}M) markedly improves the wave shape. Increase in H_2SO_4 concn. shifts the wave to more positive values and makes the Ce^{4+} - Ce^{3+} system more reversible. The wave height is proportional to the concn. of Ce^{4+} only in the presence of ICl (10^{-6}M). Cerium is determined in soln. of cerous salts after their oxidation with $NaBiO_3$. Results for 1 to 28 mg of Ce were within ±0·7%.
G. RUSSELL

3212. **Photometric determination of rare-earth elements with "salicylfluorone".** F. V. Zalkovskii and G. F. Sadova (All-Union Inst. of Mineral Raw Materials, Moscow). *Zhur. Anal. Khim.*, 1961, **16** (1), 29–31.—Salicylfluorone [2,6,7-trihydroxy-9-(*o*-hydroxyphenyl)fluorone] forms coloured compounds with rare-earth elements in a nearly neutral medium and the soln. obey Beer's law. The reagent is more selective than arsenazo I. The determination of certain individual rare-earth metals as well as their sum with salicylfluorone is possible by using sulphosalicylic acid to mask Ca and Mg.
A. BURWOOD-SMITH

3213. **Spectrographic determination of trace amounts of rare-earth elements. I.** Z. Radwan, B. Strzyżewska and J. Minczewski (Inst. of Nucl. Res., Acad. Sci., Warsaw, Poland). *Chem. Anal., Warsaw*, 1960, **5** (6), 935–949.—Combined d.c. arc - Feussner spark excitation, an argon - oxygen (4:1) atmosphere, a sifter cathode and a copper anode were used in the method proposed. Standard soln. of La and Y (as chlorides or EDTA complexes) and $PdCl_2$ (as internal standard) were evaporated on graphite powder to which 5% of NH_4NO_3 was added to reduce the exposure time. The lines measured were La 3949·1 A, Y 3950·4 A and Pd 3958·6 A. The detectability of Y was 0·2 μg as the chloride, and 0·1 μg as the EDTA complex, per 200 mg of powder. A tenfold excess of La did not affect the emission of Y, and Pd showed no buffering action.
P. BRYCH

3214. **Determination of micro amounts of iodine in elementary silicon.** V. A. Nazarenko and M. B. Shustova (Inst. of Gen. and Inorg. Chem., Acad. Sci., UkrSSR). *Zavod. Lab.*, 1961, **27** (1), 15–16.—The pink colour of a benzene soln. of iodine is sensitive to 2 to 3 μg of iodine. The sensitivity can be increased 6 times by converting the iodine to IO_3^- and treating with KI. The sample (1 or 0·5 g) of silicon is dissolved in 20 ml of 3N-NaOH and the soln. is treated with 5 ml of H_2SO_4 (1:1) and then diluted with water to 150 ml. It is then mixed with 1 ml of a soln. prepared by gentle heating of 100 ml of conc. H_2SO_4 containing 2 g of $NaNO_2$ and extracted with benzene (2 × 10 ml). The extract is washed with water and the iodine is extracted with 0·1N-NaOH (2 × 1 ml). The alkaline soln. is treated with 1·5 ml of 3N-H_2SO_4 and 0·5 ml of bromine water and boiled until the yellow colour is discharged. It is then mixed with 3 ml of water and 0·5 ml of 1% Na sulphosalicylate soln. and treated with 0·5 ml of KI soln. (4 mg per ml). The iodine is extracted by shaking with 1 ml of benzene and the colour of the upper layer is compared with that of standards. The method is suitable for determining 0·5 μg of iodine in 1 g of silicon.
G. S. SMITH

3215. **Quantitative analysis of quartz in foundry dust of respirable size by X-ray diffraction.** R. I. Higgins and P. Dewell (B.C.I.R.A., Alvechurch, Birmingham). *B.C.I.R.A. Journal*, 1961, **9** (1), 117–125.—A procedure for the routine determination of quartz by means of a 57·3-mm powder camera, fitted with a micro-focus X-ray generator, is described. The internal standard is CaF_2. The major source of error appears to be due to the inhomogeneity of the mixture of sample and internal standard, and this is discussed together with errors due to particle size and line-coincidences with graphite and mica.
J. W. O. PYEMONT

3216. **Analysis of commercial germanium dioxide.** E. Kroužek and V. Patrovský (Inst. Raw Minerals, Prague). *Chem. Průmysl*, 1961, **11** (1), 24–26.—*Determination of Ge:* (*i*) *Titrimetric procedure*—Dissolve the sample (0·1 to 0·2 g) by boiling in H_2O (100 ml), cool, dilute to 250 ml and adjust the pH to 5 with HCl or NaOH soln. Add solid catechol (3 g), mix and titrate the catechol - germanic acid potentiometrically with 0·1N-NaOH to a pH of 5 (*cf.* Wunderlich and Göhring, *Anal. Abstr.*, 1960, **7**, 2121). (*ii*) *Gravimetric procedure*—Heat a neutral soln. of the sample to 70° and add the pptg. agent dropwise (dissolve 5 g of 8-hydroxyquinoline in 5 ml of conc. HCl, dilute to 100 ml and add 16 ml of this soln. to 42 ml of conc. HCl and 42 ml of 10% ammonium molybdate soln.) (7·5 ml per 25 ml of the sample soln.). After 4 hr. filter off the pptd. molybdogermanic acid, wash the ppt. with a soln. containing 7 ml of conc. HCl and 25 ml of 2% 8-hydroxyquinoline soln. per litre, dry at 105° to 110° and weigh. Use the factor 0·0293 for calculating the results. The method is suitable for samples containing ≯1 mg of Ge, *e.g.*, after its separation by distillation. (*iii*) *Photometric procedure*—The phenylfluorone method (Cluley, *Analyst*, 1951, **76**, 523; Luke and Campbell, *Anal. Abstr.*, 1957, **4**, 65) is suitable for samples containing 0·005 to 1 mg of Ge in 50 ml. *Determination of Cl, NH_3 and As*—Ammonia is determined by the usual distillation method; Cl^- are determined mercurimetrically in an aliquot of the residue in the distillation flask; and As in a second aliquot of the residue is titrated with 0·1N-iodine in $NaHCO_3$ medium, with starch as indicator.
J. ZÝKA

3217. **Spectrochemical determination of lead in zircon for lead-alpha age measurements.** H. Rose and T. Stern (U.S. Geol. Surv., Washington 25, D.C.). *Amer. Min.*, 1960, **45** (11–12), 1243–1256.—

Full details are given of a spectrographic method for the determination of Pb in zircon by the line Pb 2833·07 A together with a two-step iron bead for calibration of the emulsion. A synthetic standard is made from zirconia, silica glass and a high-lead glass, to cover a range of concentration of 1 to 560 p.p.m. of Pb. The results obtained show a mean error of $<5\%$, compared with the results from other techniques. R. A. HOWIE

3218. **Determination of lead and zinc in low-grade ores by X-ray fluorescence spectrometry.** M. Maurette and J. Despujols (Lab. de Chim.-Phys. de la Fac. des Sci., Paris, France). *J. Chim. Phys.*, 1960, **57** (11–12), 1099—1102.—Comparison was made of a direct method with external standards and two methods with internal standards (Sr and a part of the continuous background of sample, respectively) for determining from 1 to 12% of Pb or Zn in the presence of each other and of other elements in the gangue. The lines used were, for Pb, $L\beta_1$ (first-order reflection), for Zn the doublet $K_{\alpha_1\alpha_2}$ (second-order) and for Sr the doublet $K_{\alpha_1\alpha_2}$ (first-order). In general, the errors were approx. equal to those for chemical analyses and polarography. The direct method is preferable when the concn. of Pb and Zn do not differ greatly and the gangues in the samples are similar in composition, but when the ore deposits or the concn. of Pb and Zn differ widely the procedure with Sr as internal standard ensures greater accuracy, especially when the gangue of the ore used for calibration is similar in composition to that of the samples. The method in which the sample itself acts as internal standard is unsatisfactory, as also is the application of a semi-empirical correction for absorption by other elements in the sample (*cf.* Beattie and Brissey, *Anal. Abstr.*, 1954, **1**, 2589), except for concn. of $\simeq 10\%$. W. J. BAKER

3219. **Chromatographic separation of titanium and iron by means of cationites.** I. K. Tsitovich (Cuban Agric. Inst.). *Zhur. Prikl. Khim.*, 1961, **34** (1), 218–220.—Distribution coeff. of Ti^{IV} and Fe^{III} between HCl soln. and the ion-exchange resins KU-1, KU-2 and AN-2F are determined. Cationite KU-2 gives quant. separation of Ti from Fe in proportions from 1:1000 to 1000:1, but KU-1 is unsuitable. A column (1 cm in diam. and 10 cm long) of KU-2 (H^+ form) is washed with 0·25N-HCl, then the test soln. (0·25N in HCl) is added. Titanium is eluted with 0·25N-HCl at a rate of 30 drops per min. and then the Fe is eluted with 4N-HCl. A. BURWOOD-SMITH

3220. **Photometric determination of nitrogen in titanium carbide and boride and other refractory materials.** A. Kh. Klibus and T. N. Nazarchuk (Inst. of Metallo-Ceramics and Special Alloys, Kiev). *Zhur. Anal. Khim.*, 1961, **16** (1), 79–82.—Extraction with various organic compounds of a dye formed by the thymol - hypobromite reaction for N is studied. The most intense colour is given with n-butanol. The optimum pH for the formation of indothymol is 11. To prevent pptn. of hydroxides, oxalic acid is used for Cr^{III}, and KF for Fe^{III}, Ti and V. The complexes formed do not affect the thymol - hypobromite reaction. As little as 0·01 to 1% of N in titanium carbide can be determined. *Procedure*—Dissolve 0·1 g of titanium carbide in 2·5 ml of conc. H_2SO_4 with 2 g of K_2SO_4. After dissolution transfer the soln. to a 100-ml flask and make up to vol. with water. To a 10-ml aliquot add 20% KF soln. (0·5 ml) and one drop of phenolphthalein soln. and neutralise with 8% NaOH soln. Then add 5 ml of 2% thymol soln., mix and add dropwise 5 ml of NaOBr soln. and a further 5 ml of thymol soln. After 30 min. extract the blue dye with n-butanol (3×7 ml). Filter the extract through a cotton-wool plug and make up to 25 ml with n-butanol. Mix, and measure the colour intensity. Standard soln. are prepared from $(NH_4)_2SO_4$. A. BURWOOD-SMITH

3221. **Extraction of zirconium with tri-n-butyl phosphate.** R. F. Rolf (The Dow Chemical Co., Midland, Mich., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 149–150.—The observed extraction of microgram amounts of Zr from 6N-HNO_3 soln. by tri-n-butyl phosphate (**I**) is due to the presence of small amounts of di-n-butyl phosphate (**II**) in commercial **I**, or **II** may be formed from **I** by hydrolysis with HNO_3. **II** can be used for the extractive separation of Zr from Th and U. J. P. STERN

3222. **Extraction of zirconium with di-n-butyl phosphate and direct determination in the organic phase with 1-(2-pyridylazo)-2-naphthol. Application to fluoride solutions.** R. F. Rolf (The Dow Chemical Co., Midland, Mich., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 125–128.—Traces of Zr in Al - Mg alloys are determined by treating the sample soln. (containing 10 to 65 μg of Zr) in mineral acid with 4·3% HBF_4 soln. (1 to 2 ml), complexing with $Al(NO_3)_3$, and extracting the Zr with a soln. of di-n-butyl phosphate in $CHCl_3$. The extracted Zr is complexed with 1-(2-pyridylazo)-2-naphthol and determined colorimetrically at 555 mμ. The method is accurate to within $\pm 2\%$. In the determination of 32 μg of Zr, Al (400), Mg (200), Bi (200), V (20), Ni (20), Pb (20), Mn^{II} (20), Sn^{II} (15), Mo^{VI} (5), Th (18), U (1), Ce^{III} (20), La (9), Pr (2), Nd (6), Ru^{III} (9), Fe^{III} (15), Cr^{III} (3), Ti^{IV} (2), PO_4^{3-} (7), F^- (80), SO_4^{2-} (140), oxalate (40), citrate (20) and tartrate (20 mg) do not interfere. J. P. STERN

3223. **Micro-determination of zirconium.** K. P. Stolyarov and N. N. Grigor'ev. U.S.S.R. Pat. 131,966 (20.9.1960).—Morin is added to the test soln. to form the green morin - zirconium complex, which exhibits green fluorescence on irradiation with u.v. light of wavelength 365 mμ. The soln. is titrated with NaF soln. until the fluorescence of the soln. no longer changes sharply. The end-point is found graphically by plotting vol. of titrant *vs.* intensity of fluorescence. To determine Zr in silicate rocks, add the sample ($\simeq 0{\cdot}05$ g) to 0·4 g of fused $Na_2B_4O_7$, fuse for 2 hr. with constant mixing to a clear melt, dissolve the melt by heating with 10 ml of HCl (1:1), filter from silicic acid into a 25-ml flask, wash the residue with 5 ml of hot water, and make the filtrate up to 25 ml with HCl (1:1); to 2 ml of the resulting soln. in a microtitrimeter cell add 2 drops of 0·5% ethanolic morin, and with constant stirring titrate with 0·004N-NaF to min. readings of the photo-current, and find the end-point graphically. C. D. KOPKIN

3224. **Complexometric determination of zirconium with xylenol orange as indicator.** A. E. Klygin and N. S. Kolyada. *Zavod. Lab.*, 1961, **27** (1), 23–24.—Xylenol orange can be used in H_2SO_4 soln. (0·1 to 0·6N). The material containing Zr is dissolved in a suitable manner, *e.g.*, in HCl and H_2O_2 for Zr - Al alloys, or by fusion with $NaHSO_4$ for Zr - U alloys, and an aliquot of the soln. containing between 2 and 20 mg of Zr is boiled for 10 min. with

50 ml of N-H_2SO_4 and 1·5 g of Na_2SO_4. The soln. is diluted with 80 ml of water and again boiled and titrated with 0·02M-EDTA (disodium salt) in the presence of 1 ml of 0·05% xylenol orange soln. There is no interference from Al, La, Ce, Zn, Cd, Mn, Mg, Cl^-, NO_3^- or MoO_4^{2-}, or from Fe, In, Sc, Th, Y, Ni or Co in amounts >10 mg; Bi, PO_4^{3-}, F^- and oxalate interfere. G. S. SMITH

3225. **Spectrophotometric determination of zirconium with 2,6,7-trihydroxy-9-methylfluorone.** A. K. Majumdar and C. P. Savariar (Dept. of Inorg. and Anal. Chem., Jadavpur Univ., Calcutta, India). *Z. anal. Chem.*, 1961, **178** (5), 352–356 (in English). —The use of 2,6,7-trihydroxy-9-methylfluorone (**I**) as a colorimetric reagent for Zr (0·66 to 2·5 p.p.m.) has been investigated. The rose-red Zr - **I** complex develops completely within 2 hr. at pH 1·9 to 2·2, and is stable for $\simeq$12 hr. The extinction is measured at 500 mμ against a reagent blank, and this system obeys Beer's law from 0·1 to 2·5 p.p.m. of Zr. To the sample soln. are added in succession 1 ml of gelatin soln. (0·4%), 5 ml of **I** soln. (prep. described), 3 ml of ethanol and 2 ml of Na acetate - HCl buffer (50 ml of N-Na acetate and 52·5 ml of HCl diluted with water to 250 ml). The effect of various ions is discussed. The relative error is 2·96%. B. B. BAUMINGER

3226. **Determination of radio-zirconium in solutions containing sodium.** G. J. Hunter (U.K.A.E.A., Chem. Div., Woolwich). Analytical Method AERE-AM 71, 1960. 2 pp.—In the method originally proposed (Hunter and Perkins, *Anal. Abstr.*, 1961, **8**, 1900) difficulties were encountered with fission-product soln. containing sodium. The present modification overcomes these. *Procedure*—Add an aliquot of sample soln. (final count >200 per min.) to zirconium carrier soln. (2 ml) in a 40-ml plastic centrifuge-tube. Adjust the acidity to >2M in HNO_3. Add lanthanum carrier soln. (1 ml), 10% EDTA (ammonium salt) soln. (10 ml) and a few drops of bromophenol blue soln. Add 6M-aq. NH_3 till the ppt. has dissolved and the indicator is blue. Add 10% NH_4F soln. (3 ml), stir and set aside for >10 min. Add satd. H_3BO_3 soln. (3 ml), mix, then add 20% H_3PO_4 (2 ml) and conc. HNO_3 (5 ml). Centrifuge after 2 min., discard the supernatant liquid and wash the ppt. with H_2O (10 ml). Dissolve the ppt. in 40% HF (2 ml) and 6M-HNO_3 (5 ml). Add lanthanum carrier soln. (1 ml), mix, centrifuge and discard the ppt. Isolate the Zr as $BaZrF_6$ and proceed as described in the original method. G. RUSSELL

3227. **Determination of zirconium in complex nickel alloys.** B. B. Bach and J. T. Francis (Mond Nickel Co. Ltd., Birmingham). *Metallurgia, Manchr*, 1960, **62**, 281–283.—A spectrophotometric method, with Alizarin red S, is used for the determination of Zr in concn. of 0·05 to 0·2%. Gravimetric methods are applicable only above this concn. Other colorimetric methods are also considered. The method is applicable to alloys containing Mo, Ti, Ce, Cr, Co and Al. Hafnium is normally present (1 to 3%) in commercial zirconium samples and forms a similar lake, but the absorption is only one-quarter of that given by Zr so the effect is negligible. When the Zr:Ti ratio is too low, clean separation is difficult, but satisfactory results are obtained down to 0·01% of Zr, in the presence of 3·5% of Ti. For >0·2% of Zr, the gravimetric method with *p*-bromomandelic acid is best. S.C.I. ABRTR.

3228. **Dimethylaminohydroxyphenoxazone carboxylic acid [gallocyanine] as an indicator in the complexometric determination of thorium.** S. P. Sangal and A. K. Dey (Univ. Allahabad, India). *J. Indian Chem. Soc.*, 1961, **38** (2), 75–76.—Gallocyanine (7-dimethylamino-4-hydroxy-3-oxo-3*H*-phenoxazine-1-carboxylic acid) soln. (0·02% in 5% aq. Na_2CO_3 neutralised to pH 7·0) may be used as an indicator for EDTA titrations of Th in the range 0° to 100° at pH 2·0 to 2·7. No interference is caused by Li, Na, K, Ag, Ca, Sr, Ba, Zn, Hg, Pb or Mn. N. E.

3229. **Complexometric determination of thorium using sulphodichlorohydroxydimethylfuchsonedicarboxylic acid (trisodium salt) [Chrome Azurol S] as an indicator.** S. P. Sangal and A. K. Dey (Univ. Allahabad, India). *Z. anal. Chem.*, 1961, **178** (6), 415–419 (in English).—Thorium may be titrated with EDTA (disodium salt) at pH 1·6 to 3·0, with Chrome Azurol S (C.I. Mordant Blue 29) as indicator. The sharpest end-point was given with 0·1 ml of 0·06% aq. indicator soln. per 5 ml of 0·02M-$ThCl_4$. Satisfactory titrations were possible with concn. of reactants down to 0·01M, and the effect of temp. was negligible. The interference of 38 ions has been studied; of these, Na, K, Li, Ag, Ca, Sr, Ba, Zn, Hg, Mn and NH_4^+ do not interfere. T. R. ANDREW

3230. **Determination of the thorium concentration in different materials by the use of nuclear emulsion plates.** J. Benisz, J. Koszelski and J. Stoch (Anstalt für Kernphysik u. für anal. Chem., Pädagog. Hochsch., Katowice, Poland). *Mikrochim. Acta*, 1961, (1), 59–64 (in German).—Nuclear emulsion plates (100 μ thick) are saturated with thorium soln. and developed after 6 days; the rate-determining process is the decay of ^{224}Ra to ^{220}Rn. The number of 2-, 3- and 4-pointed stars per unit area is a function of the concn. of Th; as little as 10^{-6}% can be determined. The method is also applicable to Ra and U. P. D. PARR-RICHARD

3231. **Turbidimetric micro-method for analysis of hydrazine.** M. R. F. Ashworth (Inst. Org. Chem., Univ., Saarbrücken, Germany). *Mikrochim. Acta*, 1961, (1), 5–10 (in English).—The method is based on the reduction of SeO_2 by hydrazine (**I**) in acid soln. to colloidal Se, and the influence of a colloid stabiliser, concn. of reactants, type of acid and heating time is discussed. Good results were obtained with **I** in concn. of 10^{-3} to 10^{-4}M and SeO_2 in concn. of 2×10^{-3}M, heated in 3·6N-acetic acid for 10 min. at 100° in the presence of a 0·2% aq. soln. of poly(vinylpyrrolidone). The turbidity was measured at 520 mμ, and was little affected by an excess of most lower alcohols. P. D. PARR-RICHARD

3232. **Determination of nitric oxide in a nitric oxide - nitrogen system by gas chromatography.** R. R. Sakaida, R. G. Rinker, R. F. Cuffel and W. H. Corcoran (Chem. Engng Lab., Inst. of Technol., Pasadena, Calif., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 32–34.—The separation of NO (0·1 to 1%) from N is achieved by gas chromatography in an 8-ft. stainless-steel tube (external diameter 0·25 in.; wall thickness 0·035 in.) on Davidson No. 912 silica gel (35 to 48 mesh or 48 to 60 mesh) previously dried at 150° to 200° for 20 hr. under dry He and conditioned with at least five 2·8-ml samples of NO and three 1·9-ml samples of NO_2 under the normal

operating conditions, namely, the inlet temp. at a point is 80° to 90° and the outlet temp. at a point is 65° to 70°, each controlled to within ±0·05°. To determine the NO content, the area under the NO peak measured by a dual-thermistor thermal-conductivity cell is evaluated. The standard deviation on 90 individual measurements was ±4%. Each determination takes 20 min.

J. P. STERN

3233. **Alkalimetric determination of phosphoric acid by using polyphosphates for masking interfering ions.** Č. Nedorost and J. Brzobohatý (Fosfa, Poštorná, Czechoslovakia). *Chem. Prùmysl*, 1961, **11** (2), 79–81.—Sodium hexametaphosphate forms colourless complexes with Ca, Mg, Fe and Al which do not interfere with the indicator colour-change in alkalimetric titration. *Procedure*—Heat the sample (20 g) for 2 hr. with H_2O (800 ml), with occasional shaking. Dilute with H_2O to 1 litre, filter through paper and dilute 25 ml of the filtrate with CO_2-free H_2O (60 ml). Add sodium hexametaphosphate soln. (5%) (10 ml), mixed indicator (0·1 g of dimethyl yellow, 0·07 g of methylene blue and 1 g of phenolphthalein in 50 ml of 60% ethanol) (3 to 4 drops), and, if necessary, N-H_2SO_4 till the violet-red liquid becomes green, then neutralise with 0·1N-NaOH and continue to titrate with NaOH soln. till the liquid turns red with a slight violet shade. Carry out a blank. Determinations of water-sol. phosphoric acid (as P_2O_5) in superphosphate gave a mean error of ≯±1%.

J. ZÝKA

3234. **Titrimetric analysis with chloramine T. II. The chloramine T-arsenic(III) reaction. A potentiometric study.** E. Bishop and V. J. Jennings (Washington Singer Lab., Univ., Exeter, England). *Talanta*, 1961, **8** (1), 22–33.—A detailed study of the reaction is made and limits are established within which it is quant. and analytically acceptable. Chloramine T does not react effectively in the absence of added halide. The reaction is satisfactory in the presence of Cl^- (>0·5M) and H^+ (0·5M to 5M); in the presence of Br^- at a final concn. of 0·01M the reaction is quant. at H^+ concn. between 10^{-5}M and 5M, and in the presence of I^- at a final concn. of 0·005M the results are excellent over the pH range from 4 to 9. Catalysts such as osmic acid, vanadates, molybdates or tungstates are without influence. The use of diluted titrant soln. for determining small quantities of As in low concn. does not afford any marked improvement in accuracy.

III. The chloramine T-arsenic(III) reaction. A study of visual indicators. E. Bishop and V. J. Jennings. *Ibid.*, 1961, **8** (1), 34–42.—Five reversible and five irreversible dyestuffs have been examined for their value as indicators under the conditions in which the reaction is quantitative. Indicator error, sharpness of colour change and quality of colour change are considered. The indicators examined were rosaniline hydrochloride, methyl orange, methyl red (sodium salt), Acid Bordeaux (C.I. Acid Red 17), amaranth, *p*-ethoxychrysoidine, quinoline yellow (C.I. Acid Yellow 3), *o*-dianisidine, tartrazine, 7,8-benzoflavone and starch-iodide. The results are tabulated and recommendations are made.

C. B. BAINES

3235. **Amperometric determination of antimony and thiocyanate with hypobromite.** G. S. Deshmukh and M. C. Eshwar (Chem. Dept., Benares Hindu Univ., India). *Curr. Sci.*, 1960, **29** (10), 388–389.—The oxidations of K Sb tartrate and KSCN with hypobromite are complete at pH 8·2, and require 1 and 4 atoms of O per mol., respectively. The titrations are carried out with a rotating platinum micro-electrode (Kolthoff *et al.*, *Analyst*, 1953, **78**, 405) at +0·3 V *vs.* the S.C.E. The test soln. is mixed with 10 ml of ≃0·6M-$NaHCO_3$, diluted to 50 ml and titrated with NaOBr soln. that has been standardised with $NaAsO_2$ under the same conditions; the errors for Sb (1·6 to 9·7 mg) range from −0·31 to +0·89%, and those for SCN^- (0·33 to 2·3 mg) from −0·85 to +0·58%.

R. E. E.

3236. **Determination of copper in high-purity antimony.** J. Provazník and M. Knížek (Res. Inst. A.S. Popov, Prague). *Chem. Listy*, 1961, **55** (1), 79–82.—*Procedure*—Dissolve the sample (0·5 g) in a mixture of conc. HNO_3 (1 ml), 10M-HCl (1·5 ml) and ammonium tartrate soln. (40 g of tartaric acid dissolved in 100 ml of H_2O and neutralised with 50 ml of conc. aq. NH_3) (6·5 ml) by heating on a water bath in a silica beaker. Evaporate the soln. to half its vol., transfer with H_2O into a 100-ml separating-funnel so that the vol. is 10 ml. Add EDTA (disodium salt) (**I**) soln. (dissolve 25 g of **I** in 200 ml of H_2O and 6·5 ml of conc. aq. NH_3) (10 ml), ammonium citrate (dissolve 100 g of citric acid in 75 ml of H_2O and neutralise with 105 ml of conc. aq. NH_3) (5 ml) and cresol red soln. (0·1% in 0·001M-NaOH) (2 drops) and neutralise with aq. NH_3. Add Na diethyldithiocarbamate soln. (0·1%) (5 ml) and $CHCl_3$ (3 ml), shake the mixture for 2 min. and separate the $CHCl_3$ layer (Bi remains in the aq. layer). Repeat the extraction twice, combine the extracts, dilute with $CHCl_3$ to 10 ml and measure the extinction at 436 mμ. Compare with a calibration curve. Traces of Cu in the reagents must be previously removed by extraction with $CHCl_3$ and Na diethyldithiocarbamate soln. The method is suitable for 0·5-g samples containing 0·25 to 10 μg of Cu.

J. ZÝKA

3237. **Spectrophotometric determination of bismuth with xylenol orange.** Hiroshi Onishi and Nasumi Ishiwatari (Atomic Energy Res. Inst., Tokai, Ibaraki-ken, Japan). *Bull. Chem. Soc. Japan*, 1960, **33** (11), 1581–1584.—A method for the determination of 10 to 80 μg of Bi is described. The error is ±10%. The molar extinction coefficient of Bi in 0·1N-HNO_3 soln. is $1·6 \times 10^4$ at 530 mμ; $HClO_4$ or H_2SO_4 may be used instead of HNO_3. The interference of small amounts of Zr and Th can be eliminated by masking with F^-, and that of Fe^{3+} by reduction with ascorbic acid. Lead and Tl do not interfere.

I. JONES

3238. **Solochrome violet RS as a new colorimetric reagent. II. Micro-determination of vanadium.** H. Khalifa and S. W. Bishara (Fac. of Sci., Cairo Univ., Giza, Egypt). *Z. anal. Chem.*, 1961, **178** (5), 345–352 (in English).—The coloured complex of V with Solochrome violet RS (C.I. Mordant Violet 5) (**I**) is stable for 30 min., and Beer's law is obeyed in the concn. range 0·05 to 6 p.p.m. The spectra and the effects of pH, ethanol, acetone, time, temp. and foreign ions on the formation of the V-**I** complex are studied and compared with those on the formation of the complex of V with Fast grey RA [6-hydroxy-5-nitro-1-(3-hydroxynaphthylazo)-benzene-3-sulphonic acid] previously described (Khalifa and Farag, *Anal. Abstr.*, 1958, **5**, 2178). The contrast in behaviour of the two dyes is discussed and attributed to the presence of the nitro-group in the Fast grey RA. *Procedure*—To the

neutral sample soln., containing between 0·05 and 1, 0·4 and 4, or 1 and 6 p.p.m. of V, are added in the following order (mixed after each addition): 10 ml of HNO_3 (0·1M), 1 ml of hydroxyammonium chloride soln. (10%), 1 to 5 ml of **I** (0·1%) and water to 25 ml. The colour is developed within 1 hr., and the extinction is measured at 580 mμ against a reagent blank.

B. B. BAUMINGER

3239. **Photometric determination of quadrivalent vanadium by means of Acid chrome blue K.** Yu. V. Morachevskiĭ and I. A. Tserkovnitskaya (A. A. Zhdanov Leningrad State Univ.). *Zhur. Anal. Khim.*, 1961, **16** (1), 106–107.—Low concn. of V^{IV} in silicate rocks can be determined with a sensitivity of 0·01 μg of V per ml. *Procedure*—Fuse 0·1 to 0·2 g of sample with Na_2CO_3 and KNO_3 and leach the melt with water. Filter, evaporate the filtrate to a moist salt and dissolve it in N-H_2SO_4. Make up the clear soln. to 50 ml. To an aliquot add ascorbic acid (1 ml of 3% soln.) and a few drops of formic acid, then 6 ml of acetate buffer soln. and Acid chrome blue K [7-acetamido-2-(5-chloro-2-hydroxyphenylazo)-1-naphthol-3,6-disulphonic acid, disodium salt] (3 ml of 0·1% soln.). Dilute to 50 ml with water and determine the V content photometrically. A. BURWOOD-SMITH

3240. **Spectrophotometric determination of vanadium in presence of titanium and aluminium.** L. Giuffrè and F. M. Capizzi (Polytech., Milan, Italy). *Ann. Chim., Roma*, 1960, **50** (11), 1547–1551.—A method for the determination of V, particularly in complex catalysts containing Ti and Al, consists in first oxidising with $HClO_4$ - H_2SO_4 to convert the V into VO_4^{3-}. The diluted soln. (containing 5 to 50 μg of V) is then used to oxidise the leuco-derivative of methylene blue. The colour produced is measured spectrophotometrically at 650 mμ and is directly proportional to the concn. of V.

L. A. O'NEILL

3241. **Polarographic determination of niobium, titanium and iron in tantalum metal and oxide.** D. I. Kurbatov (Inst. of Chem., Acad. Sci. USSR, Ural Branch). *Zhur. Anal. Khim.*, 1961, **16** (1), 36–38.—Small amounts of Fe, Ti and Nb (0·01 to 1%) give distinct polarograms in the presence of large amounts of Ta (99%) when $H_4P_2O_7$ is used as a supporting electrolyte. The limiting currents are proportional to concn. A method is developed for the determination of these elements in tantalum metal and oxide on a single polarogram with a relative experimental error of ±5%. *Procedure*—Dissolve 0·5 to 1 g of the metal in HF and HNO_3; with tantalum oxide the addition of HNO_3 is unnecessary. After dissolution add 20 to 30 ml of $H_4P_2O_7$ and heat to expel the HF and HNO_3. Transfer the $H_4P_2O_7$ soln. to a 100-ml flask and make up with acid so that the ratio of $H_4P_2O_7$ to H_2O in the final soln. is 2:1. Use an aliquot of this soln. for the polarographic determination of Fe and Ti with a mercury internal electrode. For the determination of Nb, make up the $H_4P_2O_7$ soln. with conc. $H_4P_2O_7$ (1·85 to 1·9). A. BURWOOD-SMITH

3242. **Quantitative determination of niobium and tantalum by the isotope-dilution method.** L. P. Alimarin and G. N. Bilimovich (V.I. Vernadskiĭ Inst. Geochem. and Anal. Chem., Acad. Sci., Moscow, USSR). *Coll. Czech. Chem. Commun.*, 1961, **26** (1), 255–261 (in Russian).—Preliminary separations of Nb and Ta from each other and from Ti and Zr can be made by pptn. with benzeneselenous acid (**I**) and by extraction with 8-hydroxyquinoline (**II**) before determination by means of the radioactive isotopes ^{95}Nb and ^{182}Ta. Niobium is separated from Zr in N-HCl containing 0·5% of citric acid and 1·5% of **I** (ammonium salt) and Ta from Zr by increasing the citric acid concn. to 1·5%; Nb is separated from Ta, Zr and Ti by extraction at pH 4·0 to 4·5 with 2% **II** soln. in $CHCl_3$.

J. W. PRICE

3243. **Mass-spectrometric study of the processes of gas evolution during the sintering of tantalum and niobium.** V. M. Amosov and V. A. Lanis (Moscow Electric Lamp Works). *Zhur. Prikl. Khim.*, 1961, **34** (1), 84–89.—Apparatus is described for the collection and analysis of gases (H, CO and N) evolved during the sintering of tantalum and niobium metal powder compacts. From the results obtained, sintering schedules are suggested.

A. BURWOOD-SMITH

3244. **Analysis of tantalum and lutetium in irradiated hafnium.** R. F. Dufour (Knolls Atomic Power Lab., Schenectady, N.Y., U.S.A.). *U.S. Atomic Energy Comm.*, Rep. KAPL-2000-11, pp. I.1–I.2.—The rare-earth metals are first separated from Ta and Hf by a CaF_2 carrier precipitation. Lutetium is determined flame-photometrically by burning the thenoyltrifluoroacetone - isobutyl methyl ketone extract of a F^--free buffered soln. in an oxy-acetylene flame. Tantalum is separated from Hf by solvent extraction with isobutyl methyl ketone, then back-extracted into dil. H_2O_2 soln. and determined colorimetrically.

NUCL. SCI. ABSTR.

3245. **Determination of oxygen in molten samples.** M. Kaše and M. Mandl (VÚHŽ, Prague). *Hutn. Listy*, 1961, **16** (1), 56–59.—Use is made of the low-pressure method (Smiley, *Anal. Chem.*, 1955, **27**, 1098; Peterson *et al.*, *Ibid.*, 1958, **30**, 1086) in which O is burnt to CO_2 with a mixture of CuO, Fe_2O_3 and kaolin (Cook and Speight, *J. Iron St. Inst.*, 1954, **176**, 252) and transferred into a trap with argon as carrier gas, where it is separated by freezing. The change in pressure during the conversion of CO_2 to the gaseous phase is measured. The error is ≯±5% and the time required for a determination is 15 min. J. ZÝKA

3246. **Determination of sulphur-35 by means of a liquid scintillation counter.** H. K. Bothe and G. Wangermann (Inst. Appl. Radioactivity, Leipzig, Germany). *Mber. dtsch. Akad. Wiss. Berlin*, 1959, **1**, 420–423 (in German).—The sample is dissolved in a liquid scintillator and the radiation is detected by means of the resulting scintillations. This method has the advantage that no changes in the physical state of the sample need be made.

CHEM. ABSTR.

3247. **Determination of thionyl chloride in the technical product.** T. Lewandowska and Z. Gwiazda (Anal. Dept., Inst. of Org. Ind., Warsaw). *Chem. Anal., Warsaw*, 1961, **6** (1), 29–35.—Thionyl chloride is hydrolysed with NaOH at pH 8 to 9 and the liberated SO_3 is determined polarographically by modification of the method of Aulenbach and Balmet (*Anal. Abstr.*, 1955, **2**, 2403). Phosphorus trichloride (<6%) and $POCl_3$ do not interfere; S_2Cl_2 lowers the SO_3 wave at $E_{\frac{1}{2}} = -0·43$ V, but a correction can be applied as a secondary wave at $E_{\frac{1}{2}} = -0·12$ V is proportional to (although not given by) S_2Cl_2. The relative error of determination is ≃±5%, improving to ±2% in the absence of S_2Cl_2. P. BRYCH

3248. **Determination of sodium chloride in sodium dithionite.** O. Kusák (Moravské Chem. Závody, Ostrava, Czechoslovakia). *Chem. Prùmysl*, 1961, **11** (1), 26–27.—*Procedure*—Add the sample (10 g) to conc. aq. NH_3 (90 ml), dilute to 50 ml, add $CuSO_4.7H_2O$ soln. (10%) (25 ml) and decompose the dithionite by boiling for 30 min. on a water bath. Cool, dilute with H_2O to 100 ml, filter, and oxidise 50 ml of the filtrate with H_2O_2 soln. (10%) (10 ml). After 5 min. pass the liquid through a column of Catex FN (H^+ form) (1·5 ml per min.) to remove Cu, wash the column with H_2O (200 ml), slightly acidify the eluate with H_2SO_4 (1:3), add sodium nitroprusside soln. (10%) as indicator and titrate the Cl^- with 0·1N-$Hg(NO_3)_2$. J. ZÝKA

3249. **Fluorimetric determination of sub-microgram amounts of selenium.** C. A. Parker and L. G. Harvey (Admiralty Materials Lab., Holton Heath, Poole, Dorset, England). *Analyst*, 1961, **86**, 54–62.—The coloured reaction product of traces of selenous acid with an excess of 3,3′-diaminobenzidine is shown to be the monopiazselenole [5-(3,4-diaminophenyl)piazselenole] (**I**) and not the dipiazselenole assumed by Cheng (*Anal. Abstr.*, 1957, **4**, 1189) and Hoste and Gillis (*Ibid.*, 1955, **2**, 1512). The absorption and fluorescence spectra of **I** have been measured and Cheng's colorimetric procedure has been adapted to the fluorimetric determination of Se in pure arsenic. The aq. soln. (30 to 35 ml) of the residue after evaporation of a soln. of the sample (up to 2·5 g) in conc. HCl and HNO_3 is treated with 2 ml each of 2·5M-formic acid, 0·1M-EDTA (disodium salt) and 3,3′-diaminobenzidine reagent (0·5 g of the tetrahydrochloride in 10 ml of O-free water), in that order, and the pH is adjusted to 2·5 with 7N-aq. NH_3. After development of the colour in the dark, the pH is adjusted to 6·8 with aq. NH_3, the selenium compound is extracted with toluene and the fluorescence of the extract is measured at 570 mμ and referred to a calibration graph. Interference does not occur with 5-mg amounts of Cr^{3+}, Mo^{6+} or Ni^{2+}, 1 mg of Te^{4+}, or 0·5 mg of V^{5+}. Strong oxidising agents must be removed. A. O. JONES

3250. **Qualitative inorganic analysis. XVII. Detection of chromate and dichromate in the presence of each other.** W. F. Jones (Sci. Dept., Victoria Inst., Worcester, England). *Mikrochim. Acta*, 1961, (1), 88–91 (in English).—Although Feigl's tests for CrO_4^{2-} and $Cr_2O_7^{2-}$ in the presence of each other (Feigl, "Spot Tests in Inorganic Analysis," 5th Ed., Elsevier Publishing Co., Amsterdam, 1958, p. 496) can be performed in soln., the reactions are strongly dependent on pH; tests on the dry material are much more sensitive. *Procedure for CrO_4^{2-} in the presence of $Cr_2O_7^{2-}$*—To the residue after evaporating 3 drops of sample soln. to dryness add 2 drops of satd. Ni dimethylglyoxime soln. in 50% ethanol; CrO_4^{2-} give a red ppt. after 2 min. The sensitivity is 20 μg of K_2CrO_4 per 1000 μg of $K_2Cr_2O_7$. *$Cr_2O_7^{2-}$ in the presence of CrO_4^{2-}*—To the dried sample add 1 drop of fresh reagent (2·5 g of KI and 2·5 g of KIO_3 in 100 ml of H_2O) and 1 drop of 1% starch soln.; $Cr_2O_7^{2-}$ give a blue colour after 5 min. The sensitivity is 30 μg of $K_2Cr_2O_7$ per 1000 μg of K_2CrO_4. P. D. PARR-RICHARD

3251. **Compounds of molybdenum with thioglycollic acid. Photometric determination of molybdenum.** A. I. Busev and Fan Chang (M.V. Lomonosov Moscow State Univ.). *Zhur. Anal. Khim.*, 1961, **16** (1), 39–50.—The specificity of the reaction of Mo^V with thioglycollic acid in a weakly acid medium (0·3M-HCl) is considerably higher than that of Mo^{VI} at pH 3 to 5, but the sensitivity of the reaction is less. A highly specific method is developed for the determination of small amounts of Mo (1 to 40 μg per ml) in alloy steels. *Procedure*—For the calibration curve, to 10 ml of sodium molybdate soln. (≯1 mg of Mo) add 1·3 ml of HCl (1:1) and 0·5M-thioglycollic acid (2 ml), make up to 25 ml with H_2O, mix thoroughly and allow to stand for 20 min. Measure the extinction at 365 or 350 mμ. For a sample of steel, dissolve ≃0·25 g in a mixture of HNO_3 (1:1) (5 ml) and H_2SO_4 (1:1) (5 ml) and evaporate to fuming; if dissolution is incomplete, add some HCl (1:1). After cooling, add 20 ml of H_2O, heat to dissolve salts, and filter, washing the residue several times with a minimum amount of H_2O, and transfer the filtrate to a 50-ml flask. Cool and make up to volume with H_2O. To an appropriate aliquot add ascorbic acid (0·2 g per 10 mg of Fe), and after a few minutes adjust the pH to 3 and then proceed as for the calibration curve. Vanadium, large amounts of Ti, rare-earth metals, Fe, Cu and W do not interfere. A. BURWOOD-SMITH

3252. **Electrochemical determination of sub-microgram quantities of molybdenum.** H. E. Palmer (General Electric Co., Hanford Atomic Products Operation, Richland, Wash., U.S.A.). *U.S. Atomic Energy Comm.*, Rep. HW-66057, 1960. 22 pp.—The method is based on the measurement of the catalytic effect of Mo in the reduction of $HClO_4$ at a mercury cathode. As little as 0·1 μg of Mo may be determined with a standard deviation of 3×10^{-3} μg. The method is specific, with V, W and Ru as the only commonly occurring interfering elements, and these can be tolerated in concn. equal to that of the Mo. NUCL. SCI. ABSTR.

3253. **Polarographic determination of traces of iron in molybdenum and molybdenum compounds.** K. Fukker and A. J. Hegedüs [Forschungsinst. für die Nachrichtentech. Industrie (HIKI), Tungsram, Budapest, Hungary]. *Mikrochim. Acta*, 1961, (1), 92–95 (in German).—The determination is made in alkaline soln. in the presence of triethanolamine (**I**); no separations are necessary except in the presence of Cu, and 5 to 100 μg of Fe per 25 ml is determined with an accuracy of ±2·2%. *Procedure*—Dissolve 0·1 g of molybdenum metal in 1 ml of mixed acid (50 ml of conc. HNO_3, 30 ml of conc. H_2SO_4 and 20 ml of H_2O), evaporate and heat strongly to expel SO_3. Add 0·5 ml of 10N-NaOH and 2 to 5 ml of H_2O, warm and acidify with a little 20% HCl, add 2·5 ml of M-**I** and re-acidify. After a few minutes add 10N-NaOH (2·5 ml) and dilute to 25 ml. Pass N for 5 min. and polarograph between 0·4 and 1·4 V. An increasing concn. of Mo affects the sensitivity, but not the validity of the method. P. D. PARR-RICHARD

3254. **Determination of tungsten in association with iron and some other elements as tris(tri-n-butylammonium) 12-tungstophosphate.** C. C. Miller an D. H. Thow (Chem. Dept., Univ., Edinburgh, Scotland). *Talanta*, 1961, **8** (1), 43–51.—For the satisfactory production of 12-tungstophosphoric acid in this determination it is necessary to acidify an alkaline test soln. containing tungstate and phosphate. The presence of other elements is considered and it is shown that Ca^{II}, Co^{II}, Cu^{II}, Pb^{II}, Ni^{II} and Sn^{IV} have no effect. Interference from Mn^{II} and small amounts of Fe^{III} is eliminated

by the addition of ascorbic acid to the re-acidified soln. Interference due to large amounts of Fe^{III} is eliminated by adsorbing tungstophosphoric acid from 6N-HCl on to a column of cellulose, which does not adsorb $FeCl_3$, but Ti^{IV}, Ta^{V}, Nb^{V}, Cr^{III}, Mo^{VI} and V^{V} interfere seriously. Complete procedures are given for the determination of W in association with Fe and W in ferrotungsten, scheelite and wolframite. C. B. BAINES

3255. **Sodium alizarin-3-sulphonate as a chromotropic reagent. I. Composition and stability of the tungsten(VI) chelate.** Samir K. Banerji and Arun K. Dey (Univ. Allahabad, India). *Z. anal. Chem.*, 1961, **179** (1), 30–37 (in English).—Sodium tungstate forms with the reagent (Alizarin red S) a red chelate which is stable at pH 3·55 to 6·0 and has an absorption max. at 470 mμ. The ratio of metal to chelating agent in the complex is 1:2.
J. W. PRICE

3256. **Complexometric determination of quadrivalent uranium with arsenazo I indicator.** A. E. Klygin, N. A. Nikol'skaya, N. S. Kolyada and D. M. Zavrazhnova. *Zhur. Anal. Khim.*, 1961, **16** (1), 110–112.—Uranium is reduced to U^{IV} with aminoiminomethanesulphinic acid (**I**) and titrated with EDTA in the presence of arsenazo I. The method is suitable for the determination of U in its salts, alloys and oxides, and in tributyl phosphate and some other solvents. *Procedure*—For the determination of U (2 to 115 mg) in a soln. of its salts, add to an aliquot 30 ml of the reducing agent (4 g of **I** dissolved in 600 ml of 0·25N-H_2SO_4), and boil for 5 to 8 min. Evaporate to a vol. of 10 to 15 ml, add 175 ml of H_2O and adjust the pH to 1·7 ± 0·1. Add 0·1% aq. arsenazo I soln. (1 ml) and titrate the U^{IV} with EDTA soln. (5×10^{-2}M) to a colour change from blue to rose. A. BURWOOD-SMITH

3257. **Complexometric titration of uranium(IV) with thoron as indicator.** P. N. Palei and Li-Yüan Hsü (V.I. Vernadskiĭ Inst. of Geochem. and Anal. Chem., Acad. Sci. USSR, Moscow). *Zhur. Anal. Khim.*, 1961, **16** (1), 51–56.—The complexometric titration of U^{IV} with EDTA at pH 1·0 to 1·8 with thoron as indicator is possible in the presence of several elements; Al, La, Cd, Zn, Ce^{3+}, Mn^{2+}, Mg, Ca, Cr^{3+}, Ti^{IV}, Ni, Pb, NaCl, $NaNO_3$ and Na_2SO_4 do not interfere; Th, Ce^{4+}, Fe^{2+} at a ratio of 10:1, Co, Cu, Bi, Hg^{2+}, V and F^- hinder the titration. To determine U^{IV} in tetrafluoride, 0·2 g is dissolved in 10 ml of N-HCl containing 1 g of $AlCl_3.6H_2O$. The soln. is made up to 50 ml with water, and to an aliquot is added 3 ml of N-HCl. After dilution of this soln. with water to 100 ml, 6 to 10 drops of thoron soln. (0·1%) are added and the soln. is titrated with 0·003M-EDTA to a colour change from rose to orange yellow. A. BURWOOD-SMITH

3258. **Amperometric titration of uranium(VI) with chromous salts.** Z. A. Gallaĭ and G. E. Kalenchuk (M.V. Lomonosov Moscow State Univ.). *Zhur. Anal. Khim.*, 1961, **16** (1), 63–67.—A method is developed for the direct amperometric titration of U^{VI} with either $CrCl_2$ in a medium of 4N-HCl or $CrSO_4$ in a medium of 4N-H_2SO_4 and with vibrating and rotating platinum micro-electrodes. There is no interference from Pb up to a ratio of U to Pb of 1:50, nor from Zr or Th up to 1:1000. Tervalent Fe in a ratio of U to Fe up to 1:50 is determined by starting the titration at a potential of 0 V and back-titrating with $CrCl_2$, then titrating another portion of soln. from 0·5 V to give the sum of the two elements. A. BURWOOD-SMITH

3259. **Coulometric titration of uranium in nitric acid solutions.** M. O. Fulda (E.I. du Pont de Nemours and Co., Savannah River Lab., Aiken, S.C., U.S.A.). *U.S. Atomic Energy Comm.*, Rep. DP-492, 1960. 14 pp.—Uranium is reduced to the quadrivalent state and titrated with electrolytically generated Ce^{4+}. The colorimetric end-point is detected automatically by a photometric technique. Interference from NO_3^- is eliminated by the addition of urea to the titration medium. The coeff. of variation for the determination of U was 3·2% for 1-mg samples and 0·3% for 100-mg samples. With the addition of urea, as much as 4·5 milli-equiv. of HNO_3 in a 1 to 3-ml sample was tolerated.
NUCL. SCI. ABSTR.

3260. **Analytical chemistry of uranium. II. Photometric determination of small amounts of uranium with bromopyrogallol red.** V. F. Luk'yanov and E. P. Duderova. *Zhur. Anal. Khim.*, 1961, **16** (1), 60–62.—*Procedure*—Dissolve 0·5 to 1·0 g of the test material by heating on a sand bath with HCl and H_2O_2, dilute with H_2O, filter and wash the residue. To the filtrate (about 100 ml) add 15 to 17 ml of EDTA (10% soln.) and adjust the pH to 7·5 to 8. Heat to boiling and then re-adjust the pH to 8 with aq. NH_3. Precipitate the U in the warm by the addition of 1-nitroso-2-naphthol (10 to 13 ml of 2% ethanolic soln.) and leave overnight. Filter and wash the ppt. carefully with cold H_2O until the washings are colourless. Dry the ppt. and bake slightly in a muffle-furnace at 550°. Dissolve the ppt. in a few ml of conc. HCl, evaporate to a few drops, add 1 or 2 drops of 10% NH_4SCN soln. and 2% EDTA soln., dropwise, to the appearance of a red colour and then add a further 10 to 20 drops. Add 5 ml of 25% hexamine soln. (pH 6·5 to 7) and 2 ml of 0·1% bromopyrogallol red soln. in 50% ethanol. Make up to 50 ml, mix thoroughly and measure the extinction in a 3-cm cell with a red filter. A. BURWOOD-SMITH

3261. **Quantitative determination of uranium by means of fluorescence in phosphate solutions.** A. V. Davydov, T. S. Dobrolyubskaya and A. A. Nemodruk. *Zhur. Anal. Khim.*, 1961, **16** (1), 68–72.—A sensitive method is developed for the determination of U by means of fluorescence in H_3PO_4 soln. Uranium is first separated from quenching admixtures (*e.g.*, Fe, Cu, Mn, Cr, Ni and Co) by complexing them with EDTA, then U is extracted with tributyl phosphate soln. in CCl_4 [with $Ca(NO_3)_2$] and re-extracted into the aq. phase with H_3PO_4 soln. The U content is determined by the fluorescence of this extract. A. BURWOOD-SMITH

3262. **Determination of uranium in zirconium ores. A modification of the stannous chloride method.** K. Bril and S. Holzer (Orquima S/A, São Paulo, Brazil). *Anal. Chem.*, 1961, **33** (1), 55–58.—Modifications to the method of Main (*Anal. Abstr.*, 1955, **2**, 341) eliminate the interference of a 40-fold excess of V. Both U^{IV} and U^{VI} are quant. reduced by $SnCl_2$ in the presence of Fe^{III} and NaF, and sharp end-points are obtained in titrations with $Ce(SO_4)_2$, with Fe^{II} - 1,10-phenanthroline as indicator. The method is applied to the determination of U in caldasite-type zirconium ores.
J. P. STERN

3263. **X-ray Rayleigh scattering method for determination of uranium in solution.** J. L. McCue, L. L. Bird, C. A. Ziegler and J. J. O'Connor (Tracerlab. Inc., Waltham, Mass., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 41–43.—An application of the method of Ziegler *et al.* (*cf. Ibid.*, 1959, **31**, 1794) is described.
E. G. CUMMINS

3264. **Spectrophotometric determination of niobium and molybdenum with 8-hydroxyquinoline in uranium-base alloys.** Kenji Motojima and Hiroshi Hashitani (Div. of Chem., Japan Atomic Energy Res. Inst., Ibaraki-ken, Japan). *Anal. Chem.*, 1961, **33** (1), 48–52.—In U - Nb alloys (containing <5% of Nb), Nb (5 to 100 μg) is determined by extraction of the complex of Nb with 8-hydroxyquinoline (**I**) from ammonium acetate - aq. NH_3 buffer of pH 5·1 into $CHCl_3$, washing the extract with a 0·05% soln. of **I** in 0·2M-ammonium carbonate - 0·05M-KCN, and measuring the extinction of the extract at 385 mμ; the concn. of Nb is determined by reference to a calibration curve. Interference by Fe for contents of Nb <1% is allowed for by measuring the extinction also at 470 mμ (Fe complex only). When Mo is present it is determined, after removal of Nb and traces of Fe from a portion of the extract with 0·05M-oxalic acid, by measuring the extinction at 380 mμ. Under the conditions indicated, Al, Cr and Mn (50 μg each) and Ta, W and Zr (200 μg each) are not extracted, while V, Ni and Cu, which are extracted, are removed by the ammonium carbonate - KCN wash.
J. P. STERN

3265. **Spectrographic determination of traces of beryllium in uranium.** P. N. Palei and E. V. Bezrogova. *Zhur. Anal. Khim.*, 1961, **16** (1), 57–59.—Down to 10^{-6}% of Be in uranium can be determined by a combined chromatographic and spectrographic method. The sample (1·5 to 2·0 g) is dissolved in HCl with addition of H_2O_2, evaporated to dryness and dissolved in 100 ml of H_2O. The soln. is adjusted to be 0·6M in HCl and then passed through a column (10 mm in diam.) of KU-2 (H^+ form) (15 to 20 g of dry resin) at a rate of 0·5 ml per min. The column is washed with 50 ml of H_2O and the Be is eluted with sulphosalicylic acid (0·02 to 0·025M) at pH 2·7 to 3. A dark-violet ring travels down the column and the eluate before this is discarded. The succeeding eluate (about 400 to 500 ml) containing the complex of U is evaporated almost to dryness, 3 to 5 ml of conc. H_2SO_4 is added to destroy the complex and the residue is heated to 700° to 800° to obtain U_3O_8. The residue is analysed spectrographically for Be. The error is ±20% and a single determination takes 40 hr.
A. BURWOOD-SMITH

3266. **Coulometric generation and back-titration of intermediate reagents at controlled potential: application to the determination of plutonium.** W. D. Shults (Anal. Chem. Div., Oak Ridge Nat. Lab., Tenn., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 15–18.—Experiments with a slightly modified ORNL Model Q-2005 automatic electronic controlled-potential coulometric titrator are described. Plutonium has been accurately determined when the ratio of the weight of Fe and/or U to Pt was ≯10 to 1.
E. G. CUMMINS

3267. **Determination of fluorine.** I. G. Izhak. U.S.S.R. Pat. 132,432 (5.10.1960).—Fluorine is determined by the extent of decolorisation of the arsenazo - Zr complex. To determine 0·01 to 0·2 mg of F per ml, prepare standards from NaF soln. (0·001 mg of F per ml) and dilute the test soln. (0·002 to 0·018 mg of F) similarly, after neutralisation if necessary. To these soln. add 15 ml of $Zr(NO_3)_4$ soln. [3 ml of $Zr(NO_3)_4$ soln. containing 0·05 mg of Zr^{4+} per ml, and 12 ml of N-HCl] and 10 ml of 0·005% arsenazo soln. Make up to 100 ml and measure the extinctions. Find the F content of the sample from a calibration curve. If the soln. contains >0·04 mg of PO_4^{3-}, add the same amount of PO_4^{3-} to the standard soln. Interference by Fe is eliminated by reduction with 1 or 2 drops of 10% ascorbic acid soln. added to sample and standards. A similar procedure is described for determining 0·01 to 1·0 mg of F per ml.
C. D. KOPKIN

3268. **Determination of fluorine in cupola slags.** H. Green (B.C.I.R.A., Alvechurch, Birmingham). *B.C.I.R.A. Journal*, 1960, **8** (6), 838–845.—The development of a rapid spectrophotometric method is described. It is based on the measurement of the transmission of a mixture of the sample soln. with a known vol. of a standard Al soln. after treatment with a mixed ferron - aluminon reagent and reference to a calibration graph of the transmission at 515 mμ *vs.* concn. of F^-. Interference from other constituents is eliminated by treatment of the sample soln. with cation-exchange resin. The recovery of F was good and results compared favourably with those by other methods.
C. H. COWPER-COLES

3269. **Rapid fluorine analysis by wide-line nuclear magnetic resonance.** H. Rubin and R. E. Swarbrick (Schlumberger Well Surveying Corp., Ridgefield Instrum. Group, Conn., U.S.A.). *Anal. Chem.*, 1961, **33** (2), 217–220.—A specific, rapid and non-destructive method is described for determining F in various fluorocarbon liquids. The content of 20-ml samples is measured with an accuracy of about 1% and the limit of detection is 12 mg of F; the content of 0·2-ml samples is measured with an accuracy of 2%, and the limit of detection is 3 mg of F. A measuring time of 1 min. is used. Non-magnetic substances do not interfere and the influence of dilution and chemical shifts in mixtures is studied.
C. B. BAINES

3270. **Coulometric - acidimetric titration of fluoride in acetic anhydride.** W. B. Mather, jun., and F. C. Anson (Inst. of Technol., Pasadena, Calif., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 132–134.—The coulometric generation of $HClO_4$ in acetic acid - acetic anhydride soln. is applied to the titration of F^- with $HClO_4$ in anhyd. soln. Since the glass electrode is slow to reach equilibrium, 95% of the $HClO_4$ required is generated and, after 5 to 10 min., further small quantities are generated at 2-min. intervals. The determination of F^- (1·5 mg) alone is accurate to within ±0·2%. Most anions interfere and must first be replaced by ClO_4^-. With a mercury indicator electrode, Cl^- and F^- can be determined in the presence of each other with accuracies >±3·5 and >±2%, respectively.
J. P. STERN

3271. **Determination of chlorine in mixtures with sulphur dioxide.** R. Sołoniewicz (Dept. of Inorg. Chem., Polytech., Łódź, Poland). *Chem. Anal., Warsaw*, 1960, **5** (6), 967–971.—*Procedure*—The test mixture is drawn through three gas wash-bottles; SO_2 is absorbed by acid $K_2Cr_2O_7$ soln. in the

first bottle and determined iodimetrically by back-titration of the excess of $K_2Cr_2O_7$. Before the determination of SO_2 the first bottle is heated to 80° for 15 min. and air is drawn through to remove Cl, which is absorbed by KI soln. in the second bottle and determined iodimetrically; $Na_2S_2O_3$ soln. in the third bottle prevents the loss of iodine from the second bottle. The accuracy is ±3% for Cl and +2% for SO_2 (after the addition of a 4% correction).
B. K.

3272. **Voltammetric chloride ion analysis.** W. N. Carson and G. R. Fountain (Knolls Atomic Power Lab., Schenectady, N.Y., U.S.A.). *U.S. Atomic Energy Comm.*, Rep. KAPL-2000-11, pp. I.20–I.26. —The basic section of the apparatus is an electrolysis cell composed of a silver cylindrical micro-electrode and a platinum cylindrical counter-electrode. A constant current in the micro-ampere range, supplied from a constant-current source, is passed through the cell. The potential of the silver electrode serves as the measure of the Cl^-. This potential is measured with the reference electrode and a vacuum voltmeter. Tests were run on synthetic samples containing known amounts of Cl^-. Results are summarised.
Nucl. Sci. Abstr.

3273. **Radiochemical determination of microgram amounts of chloride.** J. K. Johannesson (City Council Lab., Wellington, New Zealand). *Analyst*, 1961, **86**, 72.—To the slightly acid soln. (10 ml containing 30 to 300 μg of Cl^-) are added exactly 1 ml of a soln. containing 190 μg of Cl^- labelled with ^{36}Cl and then, after mixing, exactly 0·5 ml of 0·01N-$AgNO_3$. The mixture is centrifuged, the supernatant liquid is removed by suction and the AgCl ppt. is dissolved in aq. NH_3. The soln. is evaporated to dryness on a stainless-steel planchet and counted with an end-window Geiger counter (*B* counts per min.). A blank determination is made with the 190 μg of Cl^- (*A* counts per min.). The wt. of Cl^- originally present is then given by the wt. of labelled Cl^- added multiplied by $(A - B)/B$. Recovery experiments show high precision.
A. O. Jones

3274. **Potentiometric and high-frequency titration of trace amounts of chlorides and mercaptans [thiols].** N. van Meurs (Koninklijke/Shell-Lab., Amsterdam, Netherlands). *J. Electroanal. Chem.*, 1961, **2** (1), 17–24.—*Potentiometric titration*—To the sample (20 ml of aqueous soln. or a larger vol. if non-aqueous) add acetone (75 ml) and acetic acid (10 ml), and titrate with 0·001N-$AgNO_3$, which may be in H_2O, isopropyl alcohol or acetic acid, with silver and glass electrodes. For 0·5 to 10 μmoles of Cl^-, the standard deviation was 0·05 μmole. With thiols alone, use the same procedure, but, if thiols are to be titrated in the presence of Cl^- or Br^-, use aq. NH_3 (25%) (5 ml) and acetone (75 ml) as diluting solvent, and bubble N through the soln. during the titration. The standard deviation (for 1 to 5 μmoles) was 0·04 μmole. In each titration, the electrode response is sufficiently rapid for automatic titration to be used. *High-frequency titration*—Use similar solvents, but ethanol may replace acetone. With aqueous soln., increase the acetone to 100 ml. Add the 0·001N-$AgNO_3$ in portions (0·1 to 0·5 ml), recording the equilibrium current after each addition and plotting the results. A frequency of 11 Mc/s is recommended. Results are given for determinations of Cl^- in solvents and inorganic salts, and for Cl^- and thiols in oil products.
G. Russell

3275. **Electrolytic determination of micro amounts of iodide. Oxidation to iodate with electrically generated bromine.** G. Berraz and O. Delgado (Inst. de Invest., Lab. de Invest. Quím. II, Univ. Nac. del Litoral, Santa Fé). *Rev. Fac. Ing. Quím., Argentina*, 1959, **28**, 53–64.—Iodide can be titrated with bromine generated electrically at constant current. The iodide is oxidised to iodate. The pH should be between 2·8 and 4·5 and the generator current 1 mA. The oxidation is followed by means of another amperometric system containing two polarised platinum electrodes; ≃50 μg of iodide can be determined with relative errors <1%. Chloride does not interfere.
B. F. Fullan

3276. **Low-level radiochemical separation of manganese.** J. M. Matuszek, jun., and T. T. Sugihara (Jeppson Chem. Lab., Clark Univ., Worcester, Mass., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 35–36.—A method based on the extraction of tetraphenylarsonium permanganate into nitrobenzene is described. A high degree of specificity is claimed and a recovery of 87 ± 5% is reported.
E. G. Cummins

3277. **Determination of available oxygen in manganese ores by cerate oxidimetry.** D. G. Foster (Swarthmore College, Pa., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 146.—The oxidising power of manganese ores is determined by adding about twice the theoretical amount of standard 0·1N-H_3AsO_3 in H_2SO_4, 0·01M-OsO_4 catalyst (3 drops) and ferroin indicator (1 drop), boiling, and back-titrating the excess of H_3AsO_3 with 0·1N-ammonium sulphatocerate in H_2SO_4. The results on 2·5 to 16·7% of O are accurate to within ±0·16%.
J. P. Stern

3278. **Photometric method for the determination of rhenium in catalysts.** M. A. Ryashentseva and Yu. A. Afanas'eva (N. D. Zelinsky Inst. of Org. Chem., Acad. Sci. USSR, Moscow). *Zhur. Anal. Khim.*, 1961, **16** (1), 108–109.—The calibration curve is prepared with standard soln. containing 0·01 to 0·2 mg of Re per litre. A soln. containing 10 mg of Re per ml is prepared by dissolving 1 g of rhenium in 5% HNO_3 soln. (25 ml) and making up to 100 ml with water. Aliquots of the soln. with conc. HCl (10 ml), 5% thiourea soln. (10 ml) and 20% $SnCl_2$ soln. (2 ml) are made up to 50 ml with water. After 1 hr. the extinction is measured, with a blue filter. Rhenium is extracted from Re - Al_2O_3 catalysts with 5% HNO_3 soln. (25 ml), and the extract is diluted to 100 ml with water and filtered. A 5-ml aliquot of the filtrate is then treated as for the standard soln.
A. Burwood-Smith

3279. **Formaldoxime as an analytical reagent. III. Behaviour of the reagent in solutions. Colorimetric determination of iron.** Z. Marczenko and K. Kasiura (Dept. of Anal. Chem., Politechnika, Warsaw). *Chem. Anal., Warsaw*, 1961, **6** (1), 37–49. —Formaldoxime in acid and alcoholic soln. loses the ability to form metal complexes. In a molar soln. only 55% of the formaldoxime reacts with metals, and 0·05M soln. are completely inactive. Since the inactivation on dilution is rapid, relatively conc. soln. of formaldoxime should be used, and the reacting soln. should be made alkaline without delay. Both Fe^{2+} and Fe^{3+} form coloured complexes, that of Fe^{2+} is yellow - orange, probably $[Fe(CH_2NO)_6]^{4-}$, and is quickly oxidised by air to the intensely violet Fe^{3+} complex, probably

$[Fe(CH_2NO)_6]^{3-}$. A high concn. of NH_3 is essential to complete oxidation, which takes 10 to 15 min. Sulphide, 8-hydroxyquinoline and CN^- interfere, and Mn, Ni, V, Ce, Cu and Co form coloured compounds; suitable separations or masking procedures are given. The determination of Fe in water, potatoes, kaolin and brass is described. *Procedure*—To 25 ml of sample soln. add 2 ml of M-formaldoxime [add 79 g of formaldehyde soln. (38% of HCOH) to 70 g of hydroxyammonium chloride in water, and make up to 1 litre] and 10 ml of 25% aq. NH_3, and make up to 50 ml. After 15 min. measure the extinction at 534 mμ. The sensitivity is 0·09 μg of Fe per sq. cm. Beer's law is obeyed. Interference from Mn is allowed for by running a parallel test to which about 0·1 g each of hydroxyammonium chloride and KCN are added before formaldoxime. This prevents the formation of the Fe complex, and Fe is then determined from the difference in extinctions. P. BRYCH

3280. **Chromatographic determination of micro amounts of iron in high-purity materials.** E. A. Bozhevol'nov and O. A. Karakovskaya (All-Union Sci. Res. Inst. of Chem. Reagents). *Zavod. Lab.*, 1961, **27** (1), 11–12.—Silica gel (particle size 2 μ) (1 g) is moistened with 0·6 ml of a 2% soln. of 1,10-phenanthroline in ethanol and dried in air. It is then held as a column in a 1·5-mm glass tube. A buffer soln. is prepared from 3% Na acetate soln. neutralised to pH 5·5 to 6·0 by passing 100 ml mixed with 1 to 2 ml of hydroxyammonium chloride through such a column to remove Fe. To determine Fe in high-purity hydrochloric or hydrofluoric acid or water, the sample (50 g) is evaporated to dryness and the residue is mixed with 0·5 ml of the buffer soln. and passed through the column. The colour is compared with that of a scale of standards. Very small amounts (0·05 μg) of Fe can be determined. No interference is caused by a 2000-fold excess of Pb, Bi, Cr, Tl, Mn, Sb, W or Te. The presence of 20 times as much Ni, Co, Cu, Zn or Mo reduces the colour intensity somewhat.
G. S. SMITH

3281. **Determination of ferrous iron in the presence of ferric iron with bathophenanthroline.** G. F. Lee and W. Stumm (Univ. Pittsburgh, Pa.). *J. Amer. Wat. Wks Ass.*, 1960, **52** (12), 1567–1574.—The advantages of using bathophenanthroline (4,7-diphenyl-1,10-phenanthroline) in place of 1,10-phenanthroline for determining microgram amounts of Fe^{2+} in the presence of microgram amounts of Fe^{3+} are discussed. The procedure is described.
O. M. WHITTON

3282. **Volumetric determination of iron and titanium. Application to ilmenites.**—Blanchet and L. Malaprade (Fac. des Sci., Nancy, France). *Chim. Anal.*, 1960, **42** (12), 603–607.—In acid media (pH < 0) tungstoarsenic acid $[AsO_4H_3)_2(WO_3)_{18}]$ is reduced to a blue compound by Ti^{III} but not by Fe^{II}. It may be used as an indicator for the reduction of Fe^{III} to Fe^{II} by $TiCl_3$ prior to determination of Fe^{II} with Ce^{IV}, or it may be used to denote the titration of Ti^{III} in the presence of Fe^{II} before subsequent titration of Fe^{II} (*e.g.*, from the reduction of ilmenite soln. in a Jones reductor).
T. R. ANDREW

3283. **Estimation of hydrogen in pickled steel by vacuum extraction at 700° C.** K. Sachs and M. Odgers (GKN Group Res. Lab., Wolverhampton, England). *J. Iron St. Inst.*, 1960, **196** (4), 406–414.—The effect of pickling on the hydrogen absorption and ductility of steel is studied. A three-point bend test and a vacuum-extraction apparatus are briefly described. The scatter of the results for H determination, the effects thereon of plastic deformation, H_2O adsorption, the method of preparation and storage of the sample, and the presence of sulphide inclusions, and the amounts of gas evolved at varied temp. are discussed. The gas evolved at 700° contained considerable proportions of gases other than H. J. W. O. PYEMONT

3284. **Emission spectrometric determination of the gaseous elements in metals. VII. Nitrogen in steels.** Hitoshi Kamada and V. A. Fassel (Inst. for Atomic Res., Iowa State Univ., Ames, U.S.A.). *Spectrochim. Acta*, 1961, **17** (1), 121–122.—Further work on the intensity-ratio method (*cf.* Fassel *et al.*, "Proceedings of the Second International Conference on the Peaceful Uses of Atomic Energy, Geneva," Vol. 28, United Nations, New York, 1958, p. 583), in which the line pair N 8216 A - Ar 8179 A was used, has shown the Ar 8179-A band to be absent from spectrograms of d.c. carbon arcs in Ar at atm. press. It was found that the band in question is a Rowland ghost of the strong parent Ar band at 8115·3 A, and that it is peculiar to the particular grating employed. The analytical curve obtained for the line pair N 8216 A - Ar 8053 A is shown to be of more general use.
E. G. CUMMINS

3285. **Determination of boron in stainless steel and uranium dioxide.** C. F. Bush (U.K.A.E.A., Res. Group, Riverside, Chatham, Kent). Analytical Method AERE-AM 74, 1961. 7 pp.—Steel samples (≃0·25 g) are treated with H_3PO_4; samples of UO_2 (≃0·5 g) are additionally treated with H_2O_2; any insoluble residue is dissolved in aqua regia. The soln. is heated with H_3PO_4 to remove HCl and HNO_3, and is then transferred to a silica distillation apparatus (illustrated) and H_2O is removed by heating in a glycerol bath at 180°. Methyl borate is then distilled from the flask after adding methanol (80 ml). The content of B in the distillate is determined absorptiometrically with curcumin. The limit of detection is 0·2 μg of B, and the error should not exceed 5% except at the lowest part of the range; the method can be applied to samples containing up to 60 p.p.m. of B. G. J. HUNTER

3286. **Micro-analysis of iron and steel. I. Silicon. A. Photometric determination as molybdosilicic acid.** S. Meyer and O. G. Koch (Chem. Lab. der Neunkircher Eisenwerk A.-G., »Saar, Germany). *Mikrochim. Acta*, 1961, (1), 82–87 (in German).—*Procedure*—Dissolve 100 mg of steel in 10 ml of hot H_2SO_4 (1:10), oxidise with 2 ml of 20% KNO_3 soln. and boil for 2 min. Cool and dilute to 50 ml then place 25 ml (10 ml for pig iron) of this soln. in a 100(50)-ml flask containing 50(25) ml of 2% ammonium molybdate soln. After 4 min., dilute to vol. with H_2SO_4 (1:3) and measure the extinction after 5 min. in a 5-cm cell (filter S42, Zeiss Elko-II photometer). The test takes 25 min., and the deviation from theoretical values is ⩾ ±0·03%. There is a gradual reduction of colour intensity (≃2% per 5 min.), but the results are reproducible if the procedure is followed exactly.

B. Photometric determination as silicomolybdenum blue. S. Meyer and O. G. Koch. *Ibid.*, 1961, (1), 134–139 (in German).—A modified molybdenum blue method for samples containing 0·004 to 0·4% of Si is described. *Procedure*—Dissolve 100 mg of steel in 26 ml of hot acid (50 ml

of H_2O, 20 ml of conc. HCl and 6·5 ml of conc. HNO_3, diluted to 100 ml), add H_2O (40 ml) and boil for 5 min.; cool and dilute to 200 ml. To a 25-ml aliquot add 10 ml of 2·5% ammonium molybdate soln., and set aside for 20 min. Add 10 ml of 4% oxalic acid soln. and 5 ml of $(NH_4)_2SO_4.FeSO_4.6H_2O$ soln. (6 g in 50 ml of H_2O, 1 ml of 5% H_2SO_4 and H_2O to make 100 ml). Measure the extinction immediately at 810 mμ against a reference soln. prepared by adding 10 ml of oxalic acid soln., 10 ml of ammonium molybdate soln. and 5 ml of $(NH_4)_2SO_4.FeSO_4$ soln. to a second 25-ml aliquot of the sample soln. The colour is stable for 19 hr. The mean relative error is ±2·4 to 4%.

P. D. PARR-RICHARD

3287. **Absorptiometric determination of traces of lead in iron and steel.** S. Meyer and O. G. Koch (Neunkircher Eisenwerk A.-G., Neunkirchen, Saar, Germany). *Arch. Eisenhüttenw.*, 1960, **31** (12), 711–715.—A fully oxidised solution of the sample in 6N-HCl is freed from Fe by continuous extraction with ethyl ether or by manual shaking with isopropyl ether; the residual Pb is treated with dithizone in $CHCl_3$, and the extinction of the solution is determined at 520 mμ. Some loss of Pb, depending on the concn. of Fe, occurs during the extraction, especially when ethyl ether is used; hence the amount of Pb is read from a calibration graph of extinctions for synthetic mixtures of Fe and Pb in proportions that give concn. of 4 and 40 mg of Fe per ml of the initial solution. If Bi is present, it must be removed by extraction with diethylammonium diethyldithiocarbamate in CCl_4 (*cf. Analyst*, 1953, **78**, 624) or by volatilisation as $BiBr_3$. Tests on pig iron and steel show a relative error <5%. One chemist can carry out 3 or 4 determinations per day. The method is applicable to concn. of Pb of 0·0001 to 0·5%. A. R. PEARSON

3288. **Methods for the analysis of iron and steel. Part 42: Cobalt in iron and steel.** British Standards Institution (2 Park Street, London, W.1). B.S. 1121:Part 42:1961. 7 pp.—Cobalt is pptd. with 1-nitroso-2-naphthol after a zinc oxide separation and determined photometrically with nitroso-R salt.

3289. **Isolation and analysis of oxide inclusions in steel.** S. Meyer and O. G. Koch (Neunkircher Eisenwerk A.-G., Saar, Germany). *Arch. Eisenhüttenw.*, 1960, **31** (11) 651–657.—The concn. of oxide inclusions may vary radially in cylindrical specimens and the circular cell previously described (*cf.* Koch and Sundermann, *Ibid.*, 1957, **28**, 557) with axially-placed anode is therefore replaced by a rectangular cell in which the anodic surface is a cross-section of the specimen. The pptn. of $Fe(OH)_3$ during electrolysis is prevented either by flooding the surface of the electrolyte with a soln. of silicone rubber, whereby an air-excluding skin is formed, or by continuous slow circulation of the electrolyte through a cation-exchange column, which replaces Fe^{3+} by NH_4^+, or by adding a reducing agent to the electrolyte, which may contain, *e.g.*, Na citrate (5%), KBr (1·2%), KI (0·6%) and hydrazine sulphate (1%), and has an initial pH of 5·6, rising to 7·7. The electrolysis proceeds more uniformly, with less pitting, in the presence of hydrazine. For the analysis of the oxide residue, the microchemical method (*cf.* Meyer and Koch, *Anal. Abstr.*, 1959, **6**, 2169; 1960, **7**, 1014) has been replaced for routine use by a spectrographic method (*cf.* Meyer and Koch, *Anal. Abstr.*, 1960, **7**, 2734) whereby SiO_2, Al_2O_3, FeO, MnO, CuO, Cr_2O_3, CaO and MgO can be determined on amounts <50 μg with a mean variance of 6·7%. A. R. PEARSON

3290. **Separation and determination of traces of cobalt in the presence of larger amounts of copper, iron, silver and zinc.** E. Schulek and J. Laszlovszky (Inst. Inorg. and Anal. Chem., L. Eötvös Univ., Budapest). *Mikrochim. Acta*, 1961, (1), 41–45 (in German).—For the determination of traces of Co in the starting materials for the manufacture of vitamin B_{12}, the mineralised residue from the sample in 0·1N-H_2SO_4 is shaken with dithizone (CCl_4 soln.) to remove Cu and Ag, and Co, Ni and Zn are then extracted with dithizone from the weakly alkaline aq. phase; 1 μg of Co is quant. separated from 200 μg of Cu, 200 μg of Ag and 1000 μg of Fe. The catalytic effect of Co on the oxidation of alizarin derivatives by alkaline perborate is used for its determination. P. D. PARR-RICHARD

3291. **New rapid method for the determination of nickel in ferrous and ferromagnetic metals.** C. L. Luke (Bell Telephone Lab., Inc., Murray Hill, N.J., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 96–98.—Nickel (>10 mg) is pptd. as hexa-ammineperchlorate and determined volumetrically with CN^-, with an accuracy within ±0·2%. The method takes 30 min. and is suitable for the analysis of NBS steel and of nickel alloys, but Cu (>0·5 mg) and Co (>3 mg) interfere. *Procedure*—Dissolve the sample (0·1 to 0·2 g) in HNO_3 (1:1) (3 ml) and HF (1 to 3 drops), add 70% $HClO_4$ (5 ml) and evaporate to fuming. Cool, add water (20 ml), 10% citric acid (5 ml), aq. NH_3 (25 ml) and 3% H_2O_2 soln. (5 ml), and cool at 5° for 5 min. Filter under vacuum, moisten the ppt. with HCl (5 ml), and dissolve it in H_2O (45 ml). Add 10% citric acid (2 ml) and 0·5% $AgNO_3$ soln. (1 ml); just dissolve the AgCl ppt. with aq. NH_3, add 10% KI soln. and titrate with standard KCN soln. J. P. STERN

3292. **Colorimetric determination of micro amounts of copper in nickel and cobalt solutions.** I. P. Kalinkin and G. S. Semikozov (Lensovet Leningrad Tech. Inst.). *Zavod. Lab.*, 1961, **27** (1), 17–20.—To prevent interference by Ni and Co in the determination of Cu as diethyldithiocarbamate by the use of the lead complex in $CHCl_3$ soln. their concn. must be ⩾10 and 20 g per litre, respectively. Two methods for determining Cu (<2×10^{-4} %) in salts of Ni and Co are described. The sufficiently diluted soln. (10 ml) with a pH of between 3 and 4 can be extracted with a soln. (2 × 5 ml) prepared by adding a soln. (25 to 50 ml) of 0·1 g of Na diethyldithiocarbamate to a soln. (50 to 100 ml) of 0·1 g of Pb acetate, dissolving the ppt. in 250 ml of $CHCl_3$, filtering and diluting to 500 ml with $CHCl_3$. The extinction is measured at 436 mμ. Alternatively, the soln. can be extracted with the reagent soln. followed by re-extraction of the Cu into HCl and neutralisation, and then extraction of the Cu with a $CHCl_3$ soln. of the lead complex.

G. S. SMITH

3293. **Spot test for ruthenium(III) and *p*-phenylenediamine.** Anil K. Mukherji (Coates Chem. Lab., Louisiana State Univ., Baton Rouge, U.S.A.). *Mikrochim. Acta*, 1961, (1), 1–4 (in English).—To test for Ru, a drop of test soln. (pH 4 to 8) is treated with 1 drop of a fresh 1% aq. soln. of *p*-phenylenediamine (**I**); a violet colour which turns brownish black indicates the presence of Ru. Silver, Au^{3+}, CrO_4^{2-}, Fe^{3+}, Pt^{4+} and Hg^{2+} interfere; Cu^{2+} and Pd^{2+} can be masked with EDTA. The limit of

identification is 0·5 μg. A 1% soln. of Ru can similarly be used to detect **I**; most amines, including the *o*- and *m*-isomers of **I**, do not interfere. The limit of identification is 0·01 μg.

P. D. PARR-RICHARD

3294. **Spectrophotometric determination of ruthenium. Anthranilic acid as a reagent.** A. K. Majumdar and J. G. Sen Gupta (Dept. of Inorg. and Anal. Chem., Jadavpur Univ., Calcutta, India). *Z. anal. Chem.*, 1961, **178** (6), 401–406 (in English).—Anthranilic acid forms a green complex (absorption maximum at 620 mμ) with Ru^{III} at pH 5·2 to 6 which obeys Beer's law up to 20 p.p.m. of Ru (extinction = 0·76 for a 1-cm cell). In the presence of 8 p.p.m. of Ru, 12 p.p.m. of Pd, 16 p.p.m. of Th, Sr or Zr, 20 p.p.m. of U, 32 p.p.m. of Ba, Cd or Mn, or 50 p.p.m. of Ca or Mg can be tolerated; EDTA, Rh, W, Hg, Cu, Bi, As, Co, Ni, Zn and Fe interfere.

T. R. ANDREW

3295. **Use of selenourea in analysis. II. Photometric determination of ruthenium and osmium without separation by means of selenourea. The composition and stability of the ruthenium - selenourea complex.** A. T. Pilipenko and I. P. Sereda (T. G. Sherchenko Kiev State Univ.). *Zhur. Anal. Khim.*, 1961, **16** (1), 73–78.—Selenourea (**I**) forms a coloured compound with Ru exhibiting max. absorption at 760 mμ and in which the ratio of Ru^{4+} to **I** is 1:3. Ruthenium and Os in the range 2 to 60 μg per litre are determined without separation in the presence of all the platinum metals except large amounts of Pd. *Procedure*—To 25 ml of test soln. in N-HCl add 4·5 ml of conc. HCl (the concn. of the final soln. should be about 6N), 1 ml of 5M-urea and 2·0 ml of 0·5M-**I**. After 30 min. measure the extinction at 760 mμ. The colour is stable for 45 min. For the determination of Os, to 2·5 ml of test soln. in N-HCl add 0·5 ml of 5M-urea and 2 ml of 0·5M-**I** in N-HCl. After 30 min. measure the extinction at 600 mμ.

A. BURWOOD-SMITH

3296. **Spectrophotometric determination of osmium. IV. *m*-Aminobenzoic acid as a reagent.** A. K. Majumdar and J. G. Sen Gupta (Jadavpur Univ., Calcutta, India). *Z. anal. Chem.*, 1961, **179** (1), 13–18 (in English).—*m*-Aminobenzoic acid (2% aq. soln. of the sodium salt) forms a purple complex with Os^{VI} and Os^{VIII} with an absorption max. at 500 mμ. Beer's law is obeyed over the range 0·5 to 8 p.p.m. of Os. Small amounts of the following ions do not interfere—Pd^{2+}, Rh^{3+}, Ir^{4+}, W^{6+}, U^{6+}, Co^{2+}, Hg^{2+}, Mg^{2+}, Ca^{2+}, Ba^{2+}, Sr^{2+} and Th^{4+}, but Ru^{3+}, Pt^{4+}, Bi^{3+}, Cu^{2+}, Zn^{2+}, Cd^{2+}, Cr^{3+}, Al^{3+}, Mn^{2+} and Ni^{2+} interfere even at a 1:1 ratio.

J. W. PRICE

3297. **Analytical chemistry of the platinum metals. X. Determination of osmium in microgram amounts by X-ray fluorescence.** R. Neeb (Inst. Inorg. Chem., Univ., Mainz, Germany). *Z. anal. Chem.*, 1961, **179** (1), 21–29 (in German).—The halogen complexes of the platinum metals form insoluble ppt. with benzyltriphenylphosphonium chloride (**I**), tetraphenylarsonium chloride (**II**) and tetraphenylphosphonium bromide (**III**). Small amounts of Os can be concentrated from a soln. by adding 0·5 to 1 ml dropwise to the centre of a filter-pulp tablet (4 cm × 4 cm) impregnated with reagent. After drying, an area 1·5 cm × 2 cm containing the ppt. is cut out and placed in the sample holder of the spectrometer for measurement. **I** is suitable for collecting 0·5 to 50 μg of Os from N-HCl soln.; it is not suitable for soln. containing Br^-. Both **II** and **III** can be used for collecting 0·3 to 6 μg of Os from HBr soln. The Os $L\beta_1$ line is used for measurement, and the coeff. of variation for 5 μg of Os is 5%. **I** also precipitates Pt, Pd and Ir^{IV} from HCl soln. and >20 μg of these metals interferes, but Ru and Rh are not pptd. In HBr soln. all the platinum metals are pptd. with **II** and **III** and interfere similarly; Ru up to 10 μg is without effect. Platinum, Pd and Ir can be determined in a similar manner, but Rh is not completely pptd. by the reagents and so cannot be determined.

J. W. PRICE

3298. **Investigations with iridium-192 of separations of platinum and rhodium from iridium. II.** K. W. Lloyd and D. F. C. Morris (Brunel Coll. of Technol., London, W.3, England). *Talanta*, 1961, **8** (1), 16–21.—Milligram amounts of Pt are separated from Ir in soln. in 10% HCl (50 ml) by means of either solid HgCl, an aq. slurry of HgCl or a mixture of 5 ml of 3% $HgCl_2$ soln. and 5 ml of 10% H_3PO_3 soln. In each case the resulting soln. is boiled for 10 min. and the pptd. Pt is collected and ignited to give the pure metal. Rhodium is separated from Ir by means of a slurry of HgCl in the presence of KBr. The pptd. Rh is collected and ignited, and finally reduced in hydrogen. Iridium is pptd. from the filtrate by the addition of KI and a further quantity of HgCl slurry. A new rapid procedure for the separation of Rh and Ir and their subsequent determination is described.

C. B. BAINES

See also Abstracts—**3395**, ^{35}S in biol. materials. **3401**, **3402**, U in urine. **3404**, Ra in urine. **3486**, CO in air. **3487**, Fluoride in air. **3495**, Be in mineral water. **3496**, U in sea water. **3536**, Micro-titrimeter for Fe. **3538**, Fluorescence apparatus for U determination. **3552**, Neutron activation in geochemistry. **3553**, Neutron absorptiometry of rare-earth metals. **3555**, Nuclear magnetic resonance in study of metals.

3.—ORGANIC ANALYSIS

Determination of elements and radicals and of organic compounds not included in other sections. Organic industrial products, including petroleum and its products, fuels, detergents, volatile oils, cosmetics, dyestuffs, fibres, plastics, resins, paints, elastomers, leather and explosives.

3299. **Sub-micro methods for the analysis of organic compounds. XIII. The cryoscopic determination of molecular weight.** R. Belcher, M. Sobotka and W. I. Stephen (Chem. Dept., The Univ., Birmingham, England). *J. Chem. Soc.*, 1961, 480–482.—The cryoscopic constant for camphor is determined by transferring ≃1 mg of camphor and a 15-mm length of platinum wire to a capillary tube (50 mm × 2 mm at the open end and 0·5 to 0·6 mm at the sealed end) and sealing about 2 mm above the wire. Another tube that has been weighed on a micro-balance is similarly charged with camphor and re-weighed. Acetanilide (30 to 96 μg) (as sample) is transferred to a weighed platinum wire by touching the wire against a melted crystal in a platinum cup (a depression in a piece of foil). The wire is re-weighed on a sub-micro balance and placed in the capillary, which is then sealed 2 mm above the wire. The tubes are placed in a m.p. apparatus at 175° and heated to 5°

above the m.p. of the contents. The current to the element is then reduced until the tubes cool at 1° per 20 sec., and the temp. at which crystals first appear is read within 0·1°. In the sample tube during the melting process the sealed-in platinum wire is allowed to move about and mix the contents thoroughly. The results for a number of org. compounds are given. E. J. H. BIRCH

3300. **Applications of mass spectrometry to organic analysis.** C. Bokhoven (Centr. Lab., Staatsmijnen in Limburg, Geleen, Netherlands). *Chem. Weekbl.*, 1960, **56** (50), 693–700.—The principles of mass spectrometry are given. The following applications are dealt with: (*i*) determination of molecular weight (*e.g.*, of 9,9′-dianthracene and cholestane), (*ii*) determination of elemental composition, (*iii*) identification of traces of impurities (*e.g.*, impurities in cyclohexanol), (*iv*) elucidation of certain details in the structure (*e.g.*, determination of the length of the side chains in steroids and the distinction between 2-phenyl- and 3-phenyl-hexane). M. J. MAURICE

3301. **Identification of organic compounds by gas chromatography.** I. Brown (C.S.I.R.O., Melbourne, Australia). *Nature*, 1960, **188**, 1021–1022.—A method has been developed for the identification of compounds by using three stationary phases: one non-polar, one an electron donor and one an electron acceptor. Retention times were measured for a number of substances at 125° on three 2·5-metre columns, each containing 1·5 g of stationary phase on 9 g of 'Celite 545,' and with a nitrogen-inlet pressure of 1000 torr above the outlet pressure (atmospheric). The three stationary phases used were 'Apiezon L,' 'Reoplex 400' (a polyester) (electron donor), and 1,3,5-trinitrobenzene (electron acceptor). Data for a particular substance are plotted as points on a triangular graph using an "affinity fraction," A, of the substance for each phase. A for phase 1 is defined as $A_1 = r_1/(r_1 + r_2 + r_3)$ where r_1, r_2 and r_3 are the retention times (or volumes) of a substance on the three stationary phases 1, 2 and 3, under fixed conditions. Each compound has a position on the graph governed by its electron acceptor - donor properties. A triangular graph is given showing the position of some 40 organic compounds when the above system is used. The method has been used for identification of coal-tar components and should prove useful for other degradation systems. R. A. HENDEY

3302. **Micro-analysis of organomercury compounds. I. Determination of carbon, hydrogen and mercury in organic compounds that do not contain halogens.** A. I. Lebedeva and E. F. Fedorova (Inst. of High-Mol.-Weight Compounds, Acad. Sci. USSR, Leningrad). *Zhur. Anal. Khim.*, 1961, **16** (1), 87–90.—Organomercury compounds are burnt in a current of O in a quartz tube containing the product of the thermal decomposition of $AgMnO_4$ heated at 60°. Carbon and H are determined with errors $\gtrsim \pm 0{\cdot}3\%$. A rapid micro-method is developed for the determination of Hg in organomercury compounds that do not contain halogens by burning them, absorbing the reaction products in conc. HNO_3 and titrating the Hg^{2+} formed with 0·1N-NH_4SCN, with $(NH_4)_2SO_4.Fe_2(SO_4)_3$ as indicator. The error is $\pm 0{\cdot}5\%$. A. BURWOOD-SMITH

3303. **Use of conductimetry in the direct micro-determination of oxygen in organic compounds.** N. É. Gel'man, Wên'-Yün Wang and I. I. Bryushkova (Inst. of Element.-Organic Compounds, Acad. Sci., USSR). *Zavod. Lab.*, 1961, **27** (1), 24–28.—By means of a rapid flow of the inert gas, a greater length of catalyst, automatic movement of the furnace and measurement of the conductivity of the alkali absorbent of the CO_2, the determination of O in organic compounds can be carried out in 10 min. The max. error in the determination of O in 13 compounds was $\pm 0{\cdot}3\%$ of the sample wt. G. S. SMITH

3304. **The oxygen flask method. A review.** A. M. G. Macdonald (Dept. of Chem., The Univ. of Birmingham, England). *Analyst*, 1961, **86**, 3–12.—After an historical introduction, the apparatus and general procedure used in the oxygen flask method for the combustion of samples are described. The appropriate absorption soln. for the products of combustion are considered and methods of completion of the determination of Cl, Br, I, S, F, P, As, B, C and metals are described. (62 references.) A. O. JONES

3305. **Micro-analytical apparatus for the determination of nitrogen.** Loughborough Glass Co., Ltd. [Inventor: I. K. H. Otter]. Brit. Pat. 862,674; date appl. 13.6.58.—The N of the sample is obtained as a gas by heating the compound with copper oxide in the presence of a stream of CO_2 and bringing the resulting gases into contact with reduced copper metal. The apparatus used for the combustion comprises two concentric tubes of quartz glass. The outer tube is sealed at one end, and the inner tube, which enters the outer tube at the other end, terminates short of the sealed end and is provided with a side-arm, at a point remote from the end nearer the sealed end of the outer tube, which passes through the wall of the outer tube. The annular space between the concentric tubes is filled with copper oxide wire, and a portion of this oxide is reduced to copper metal in a stream of H before the combustion of the sample. The N evolved is measured in a nitrometer. J. M. JACOBS

3306. **Potentiometric finish in the Schöniger halogen method.** D. G. Newman and C. Tomlinson (Microanal. Lab., Dept. of Org. Chem., Liverpool Univ., England). *Mikrochim. Acta*, 1961, (1), 73–74 (in English).—A modified combustion flask which allows for electrode insertion and magnetic stirring is described. Direct handling of the sample paper leads to contamination with Cl^- and should be avoided. After being boiled for 2 min. with 1 mg of platinised asbestos to destroy the last traces of H_2O_2, the absorbing soln. is acidified with 9N-H_2SO_4 against *p*-bromothymol blue, re-boiled and cooled, and diluted 1:1 with ethanol. This soln. is titrated with 0·01N-$AgNO_3$, while being stirred magnetically; silver and Hg_2SO_4 electrodes are used. The standard deviation for acet-*p*-chloroanilide was 0·19 (18 determinations). Minor modifications are required for determination of Br^-. P. D. PARR-RICHARD

3307. **Studies in organoselenium compounds. I. Determination of selenium.** D. Dingwall and W. D. Williams (Sch. of Pharmacy, Royal Coll. of Sci. and Technol., Glasgow). *J. Pharm. Pharmacol.*, 1961, **13** (1), 12–19.—Selenium is determined colorimetrically by conversion into selenous acid and thence into a Se sol stabilised by chlorpromazine hydrochloride (**I**). Non-ionic, cationic and anionic stabilisers have been compared with **I** but only cetomacrogol 1000 appears to be as efficient.

Procedure—Dissolve the sample (containing 2 to 3 mg of Se) in conc. H_2SO_4 (1 ml) and conc. HNO_3 (2 ml) in a Kjeldahl flask and evaporate to dryness. If the residue is red or brown, add HNO_3 (one drop) and remove the excess by heating. Cool the residue, dilute with H_2O (10 ml) and neutralise to phenolphthalein with 20% NaOH soln. Cool to 20°, dilute to 25 ml with H_2O and mix. To a 5-ml aliquot add conc. HCl (one drop), 2% **I** soln. (1 ml), H_2O (2 ml, if needed to give a clear soln.) and 1% ascorbic acid soln. (1 ml). Dilute with H_2O to 10 ml, mix, set aside for 30 min. and measure the extinction at 420 mμ against a reagent blank. The calibration curve, prepared from known amounts of pure colourless crystalline SeO_2 (<1 mg) in the presence of Na_2SO_4 (0·5 g) and conc. HCl (one drop) in a final vol. of 10 ml, is linear.

A. R. Rogers

3308. **Volumetric determination of phosphorus in organic compounds.** E. Meier (Weizmann Inst. of Sci., Microanal. Lab., Rehovoth, Israel). *Mikrochim. Acta*, 1961, (1), 70–72 (in German).—After a Schöniger flask combustion of the sample (2 to 6 mg), absorb the gases in H_2O (20 ml) and 30% H_2O_2 soln. (1 ml) and boil the soln. for 30 min. Dilute the boiled soln. (≯20 ml) to 60 ml, and neutralise to phenolphthalein with aq. NH_3 soln., then discharge the colour with 1 drop of HNO_3 soln. (1:20). Add 1 or 2 drops of Eriochrome black T soln. and an excess of 0·01N-$Pb(NO_3)_2$, and back-titrate with 0·01N-KH_2PO_4. If S is present, dilute the original soln. to 50 ml and determine S in an aliquot by titration with 0·02N-$Ba(ClO_4)_2$ in isopropyl alcohol soln., adding a mixture of 3 drops each of aq. methylene blue soln. (0·015%) and aq. thoron soln. (0·2%) and 60 to 80 ml of isopropyl alcohol. Results for P are 3 to 5% low.

P. D. Parr-Richard

3309. **Elementary organic analysis by the "wet combustion" method. II. Rapid determination of silicon in organic silicon compounds.** A. P. Terent'ev, S. V. Syavtsillo and B. M. Luskina (M. V. Lomonosov Moscow State Univ.). *Zhur. Anal. Khim.*, 1961, **16** (1), 83–86.—A rapid method for the determination of Si in organic silicon compounds is based on their oxidation by a mixture of chromic and sulphuric acids at 150°. The resulting SiO_2 is filtered off, dissolved in NaOH, and converted into a fluorosilicate ion by ammonium fluoride in the presence of an acid; the excess of acid is back-titrated with alkali. The determination takes 1·5 hr., but when the silicon compound is readily hydrolysable with an aq. alkali soln. with the formation of SiO_2 the determination time is 30 min.

A. Burwood-Smith

3310. **Use of magnesium metal in the determination of alkali metals.** P. N. Fedoseev and M. E. Grigorenko (S.O. Makarov Nikolayev Shipbuilding Inst.). *Zhur. Anal. Khim.*, 1961, **16** (1), 100–102.—*Procedure*—Place 0·05 to 0·3 g of the organic test substance in a stainless-steel vessel (200 mm by 10 to 12 mm in diam.) containing magnesium powder (40 to 50 mg), then add a little more magnesium (400 mg) and mix carefully. To prevent the formation of magnesium nitride add a few drops of ethyl ether (0·5 to 1 ml). Heat the mixture for 20 to 25 min. at 650° to 700°. On cooling add 50% ethanol (7 to 10 ml) and mix with a metal stirrer. Filter and wash the residue with ethanol, keeping the vol. down to 120 to 150 ml. Determine the alkali metals in the filtrate by back-titrating with 0·1N-H_2SO_4 in the presence of methyl red. For a gravimetric determination, evaporate the titrated soln. to 5 to 10 ml, transfer to a platinum crucible, acidify with 2 or 3 drops of conc. H_2SO_4 and evaporate to dryness. Heat in a muffle-furnace at 600° to 650° and weigh the alkali-metal sulphates.

A. Burwood-Smith

3311. **Applications of infra-red spectroscopy. III. The simultaneous determination of methoxyl and ethoxyl groups.** D. M. W. Anderson and J. L. Duncan (Dept. of Chem., Univ., Edinburgh, Scotland). *Talanta*, 1961, **8** (1), 1–7.—The method described is accurate, rapid and superior to any previous method in that small samples are required, scrubbers are eliminated and sulphur in any form or amount does not interfere. The alkoxyl groups are determined as alkyl iodides when the methoxyl to ethoxyl ratio is <4:1, and as alkyl bromides when it is >4:1. The reaction time is 30 min. and results, correct to within ±1% for each alkoxyl group, can be obtained on the micro-scale. The apparatus is described (*cf. Ibid.*, 1960, **7**, 70).

C. B. Baines

3312. **Determination of the oxidation products of low-molecular hydrocarbons by means of gas chromatography.** Yu. D. Norikov (Inst. of Chem. Physics, Acad. Sci., USSR). *Zavod. Lab.*, 1961, **27** (1), 28–30.—The method is based on the use of triethylene glycol as stationary phase and a thermal-conductivity detector.

G. S. Smith

3313. **Determination of 3-bromo-1-chloropropane in the presence of the 2,1-isomer by use of sodium iodide in acetone.** I. Krasznai and Z. Tóth (United Pharm. and Health-food Works, Budapest). *Magyar Kém. Foly.*, 1961, **67** (1), 36–40.—Experiments showed that, under favourable conditions of temp. (26°) and reaction time (14 to 16 hr.), the 2,1- and 3,1-isomers of bromochloropropane undergo an exchange of Br with I with NaI dissolved in acetone to widely differing extents. The 2,1-isomer reacts to give NaBr in very low yield (0·35%), while the 3,1-isomer reacts virtually quantitatively. The ppt. obtained with the 3,1-isomer has the average composition 2·82% of NaI, 0·48% of NaCl and 96·43% of NaBr. The following method is based on these observations. The sample of mixed isomers is dissolved in acetone (≃1·8 g per 100 ml), and 20 ml of this soln. is treated with 20 ml of 15% NaI soln. in acetone under the reaction conditions specified above. The ppt. so obtained is collected, washed with acetone, dried (105°) and dissolved in water. The soln. is then titrated by the Volhard procedure; 1 ml of 0·1N-$AgNO_3$ ≡ 15·75 mg of 3-bromo-1-chloropropane. The error of the determination is up to ±1·7%. Water and organic compounds that contain replaceable Cl or Br interfere.

B. Vaszko

3314. **Failure of alkaline methods to measure degree of polymerisation of carbohydrates. Comparison with chlorite method.** H. F. Launer and Yoshio Tomimatsu (W. Reg. Res. Lab., Albany, Calif., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 79–83.—The chlorite method was compared with those involving hypoiodate, copper and ferricyanide in the presence of alkali. In general, the alkaline methods failed to measure the aldehyde content, whereas the chlorite method gave values that agreed closely with the results of other methods when applied to the same series of periodate starches.

G. P. Cook

3315. **Differential thermograms of polysaccharides.** G. Chesters and S. O. Thompson (Dept. of Soils, Univ. Wisconsin, Madison, U.S.A.). *Science*, **1961**, **133**, 275–276.—The thermo-analysis of carrageenans and various other polysaccharides has been studied by using a method similar to that of Morita (*Anal. Chem.*, 1956, **28**, 64). Some closely related polysaccharides, *e.g.*, amylose and amylopectin, showed widely different thermal behaviour. It is suggested that this technique may be used for polysaccharide characterisation and for general identification of organic compounds.
R. A. HENDEY

3316. **A new reagent for the characterisation of aldehydes: (±)-1,2-dianilino-1,2-diphenylethane.** R. Jaunin and J.-P. Godat (Lab. Chim. Org., Univ., Lausanne). *Helv. Chim. Acta*, 1961, **44** (1), 95–98 (in French).—The reagent (**I**) (Jaunin, *Ibid.*, 1956, **39**, 111) serves to detect aldehydes in the presence of ketones. Under acid conditions, it reacts with most aliphatic aldehydes to give (±)-1,3,4,5-tetraphenylimidazolidines (**II**), which have sharp melting-points; choral, trimethylacetal, citral and glucose do not react. Only in favourable cases (halo- or nitro-derivatives) do aromatic aldehydes react. The mol. wt. of the **II** may be determined by potentiometric titration. *Preparation of* **II**—Dissolve **I** (1 g ≡ 2·75 millimoles) in acetone (25 ml), add the aldehyde (3 millimoles) and a soln. of H_2SO_4 (0·1N) in glacial acetic acid (25 ml). Allow to stand for 2 to 30 min. Add enough H_2O to obtain a temporary turbidity and induce crystallisation by scratching the sides of the vessel. Allow to stand for 30 min. and add H_2O (≃ 50 ml) slowly. Allow to stand for 10 to 12 hr.; wash the ppt. well and dry. The yield is 95 to 100%. Recrystallise from ethanol or ethanol - ethyl acetate. For the titration, dissolve the **II** (150 to 250 mg) in dry benzene (0·5 to 2 ml), add acetic anhydride (≃50 ml) and titrate with $HClO_4$ (0·1N) in glacial acetic acid; 1 equiv. of **II** ≡ 1 equiv. of acid. Results are given for 28 aldehydes. G. RUSSELL

3317. **The 3-methyl-2-benzothiazolinone test. Sensitive new methods for the detection, rapid estimation and determination of aliphatic aldehydes.** E. Sawicki, T. R. Hauser, T. W. Stanley and W. Elbert (Robert A. Taft Sanitary Engng Center, U.S. Dept. Health, Educ. and Welfare, Cincinnati, Ohio). *Anal. Chem.*, 1961, **33** (1), 93–96.—The methods are based on the reaction of the aldehyde with 3-methyl-2-benzothiazolinone to form an azine, which is oxidised to a blue cationic dye. The extinction is measured at either 635 or 670 mμ depending upon whether the dye is in aq. or acetone soln., and Beer's law is obeyed in the range from 5 to at least 125 μg of aldehyde per 100 ml of soln. The detection of as little as 0·1 μg can be carried out on a spot-plate or filter-paper or in a silica-gel tube, and can be applied to the examination of polluted air. G. P. COOK

3318. **Simultaneous determination of formaldehyde and acetaldehyde in the pentaerythritol production process.** D. Sikorska and K. Hetnarska (Anal. Dept., Inst. of Org. Ind., Warsaw, Poland). *Chem. Anal., Warsaw*, 1960, **5** (6), 1063–1068.—*Procedure*—N-Hydroxyammonium chloride is added to the test soln. containing formaldehyde (**I**) and acetaldehyde (**II**) and after 30 min. the liberated HCl is titrated with 0·1N-NaOH. From the sum of **I** and **II** so obtained the amount of **I**, determined polarographically, is subtracted. The error is ±0·9% for **I**, and ±0·8% for **II**. Pentaerythritol and $Ca(OH)_2$ do not interfere. B. K.

3319. **Determination of paraformaldehyde by the hydroxylamine method with sulphuric acid as the depolymerisation agent.** S. Chwaliński and W. Skorupski (II Chem. Dept., Polytech., Warsaw, Poland). *Chem. Anal., Warsaw*, 1960, **5** (6), 1005–1009.—*Procedure*—Dissolve the sample of paraformaldehyde (**I**) in H_2SO_4 soln., neutralise with N-NaOH, add 0·5N-hydroxylamine (previously neutralised with 0·1N-NaOH), mix and set aside. After 15 min. titrate the liberated HCl with 0·1N-NaOH. Various concn. of H_2SO_4 were tested. It was found that H_2SO_4 (1 : 1) is the best medium for the depolymerisation of **I**. Lower concn. of H_2SO_4 prolong the time required for the determination. The accuracy is the same as that of the classical iodimetric method. B. K.

3320. **Simultaneous determination of acetaldehyde and paraldehyde.** Z. Bellen and B. Sękowska (Struszyński Anal. Lab., Inst. of Gen. Chem., Warsaw). *Chem. Anal., Warsaw*, 1961, **6** (1), 69–74.—Acetaldehyde is oxidised to acetic acid by ammoniacal silver soln., and the excess of Ag^+ is back-titrated. Paraldehyde is depolymerised by acid treatment and total acetaldehyde is distilled off into ammoniacal silver soln. *Procedure for acetaldehyde*—To 50 ml of 0·01N-$AgNO_3$ add 1 ml of 6N-NaOH and dissolve the ppt. in a few drops of conc. aq. NH_3. Add the sample containing 20 to 80 mg of acetaldehyde, stopper the flask and digest the contents for 15 min. Then titrate the excess of Ag^+ potentiometrically with KI soln. or complete by a Volhard titration. *Acetaldehyde and paraldehyde*—Add the sample containing 20 to 80 mg of total aldehyde to 100 ml of water, acidify with 2 ml of conc. H_2SO_4 and distil the acetaldehyde into the ammoniacal silver soln. prepared as described above. Collect about 80 ml of the distillate, digest for 15 min. and complete as described above. The relative error is $< \pm 1\%$.
P. BRYCH

3321. **Chromatographic behaviour of 2,4-dinitrophenylhydrazones on chromatoplates.** J. H. Dhont and C. de Rooy (Central Inst. for Nutrition and Food Res. T.N.O., Utrecht, Netherlands). *Analyst*, 1960, **86**, 74–76.—In an investigation of volatile carbonyl compounds in foods, 2,4-dinitrophenylhydrazones (**I**) were conveniently separated on chromatoplates. These are prepared by means of a simple apparatus described by Stahl (*Pharmazie*, 1956, **11**, 633) in which glass plates are coated with a film of a standardised mixture of silicic acid and gypsum (Silica gel G). The test substance (dissolved in $CHCl_3$ or dioxan) is spotted on to the plate, previously activated by heating at 110° for 5 min., and chromatographed by the ascending technique with benzene - light petroleum (3 : 1) for aliphatic **I** and benzene containing 5% of ethyl acetate for aromatic **I**. The activity of the plate is calibrated by means of a test mixture of butter yellow (*p*-dimethylaminoazobenzene) (**II**), indophenol blue and Sudan red G (C.I. Solvent Red 1) in a volatile solvent, the R_B value being defined as the ratio of the ascent of **I** to that of **II**. With some mixtures of vanillins with other carbonyl compounds, two-dimensional chromatography with the two solvents is advisable. The spots can be scraped from the plates and the dinitrophenylhydrazones extracted

with $CHCl_3$ and subjected to u.v. spectrophotometry, m.p. determination and flash-exchange chromatography (Ralls, *Anal. Abstr.*, 1960, **7**, 4825). A. O. JONES

3322. **Spectrophotometric studies of the 2,4-dinitrophenylhydrazones of n-alka-2-enals and n-alka-2,4-dienals.** F. Stitt, R. B. Seligman, F. E. Resnik, E. Gong, E. L. Pippen and D. A. Forss (Western Reg. Res. Lab., Agric. Res. Service, U.S. Dept. of Agric., Albany, Calif.). *Spectrochim. Acta*, 1961, **17** (1), 51–63.—Wavelength maxima of 357, 375 and 391 mμ are reported for the 2,4-dinitrophenylhydrazine derivatives of n-alkanals, n-alka-2-enals, and n-alka-2,4-dienals, respectively, in $CHCl_3$ soln. In alkaline ethanol soln. the corresponding figures are 430, 458 and 480 mμ. It is noted that in n-hexane soln. the respective values are 337, 356 and 374 mμ. Differences in the infrared spectra which likewise depend on the degree of unsaturation are discussed. E. G. CUMMINS

3323. **Chloranilic acid as a reagent in the paper chromatography of the sodium salts of organic acids.** H. S. R. Barreto and R. C. R. Barreto (Nat. Sch. of Agric., Rural Univ., Rio de Janeiro, Brazil). *J. Chromatography*, 1961, **5** (1), 1–4 (in English).—Chloranilic acid (**I**) is used to detect the sodium salts of organic acids separated by paper chromatography with propanol - aq. NH_3 (7:3) as solvent. The thoroughly dried chromatogram is dipped in a 0·1% soln. of **I** in ethyl ether, washed twice in ether, dried and observed under u.v. light. Amounts as small as 0·2 μg of Na can be detected; the sensitivity to Na nicotinate and Na *p*-aminobenzoate is greater than expected. A. R. ROGERS

3324. **Iodimetric estimation of formates by oxidation with iodine.** R. M. Verma and S. Bose (Department of Chemistry, Mahakoshal Mahavidyalaya, Jabalpur). *J. Indian Chem. Soc.*, 1961, **38** (2), 109–112.—A neutral soln. of formate (30 to 90 mg in 10 to 20 ml) is treated with 20 ml 0·0125M- KIO_3 and 10 ml of 0·4N-iodine and kept in boiling water for 30 min. After cooling, the soln. is titrated with 0·1N-$Na_2S_2O_3$, then acidified with 2 ml of N-H_2SO_4 and the liberated iodine is titrated. A blank is carried out. The difference between the two titrations × 4·6 ≡ mg of HCOOH. N. E.

3325. **Paper-chromatographic separation and identification of the C_4 to C_{14} fatty acids as their 2,4-dinitrobenzyl esters. II.** J. Churáček (Inst. für anal. Chem., Chem. Technol. Hochsch., Pardubice, Czechoslovakia). *Mikrochim. Acta*, 1961, (1), 65–69 (in German).—The esters are prepared by dissolving the sodium salts of the acids in a ternary mixture of benzene, ethanol and H_2O and treating the soln. with 2,4-dinitrobenzyl bromide at 100° for 1 hr. in a sealed tube; 2 to 6 μl of the reaction mixture is used for the separation on dimethylformamide-impregnated paper. The chromatogram is developed with light petroleum (190° to 200°) - cyclohexane (1:35), and Na ethoxide soln. in acetone is used as a detecting reagent. A list of R_F values is given. P. D. PARR-RICHARD

3326. **The application of bromine chloride in analysis. Determination of maleic and fumaric acids.** K. Burger and E. Schulek (Inst. for Inorg. and Anal. Chem., L. Eötvös Sci. Univ., Budapest). *Magyar Kém. Foly.*, 1961, **67** (1), 33–36.—The method proposed is based on the finding that the reaction of BrCl with maleic and fumaric acids in the presence of Hg^{2+} is complete in 15 min. The soln. containing the maleic or fumaric acid is measured into a glass stoppered flask, and sufficient 0·5M-$HgSO_4$ to combine with all the chlorine is added, followed quickly by a known amount of 0·1N-BrCl (20 to 120% in excess of theoretical requirements). The soln. becomes almost colourless on shaking. After 15 min. KI soln. is added until the HgI_2 ppt. dissolves. The iodine equivalent to the excess of BrCl is then titrated with 0·1N-$Na_2S_2O_3$, with starch as indicator; 1 ml of 0·1N-BrCl ≡ 5·8035 mg of maleic or fumaric acid. The error of the determination is ±1%. For the determination of maleic acid in the presence of fumaric acid, 15 to 20 ml of soln. containing 2 to 5 mg of maleic acid and ⩾6 mg of fumaric acid is neutralised to methyl red indicator; 0·5M-$HgSO_4$ (5 drops) is then added and the resulting soln. is allowed to stand for 1 hr. before being centrifuged. The pptd. mercury fumarate is filtered off and the maleic acid content of the filtrate is determined with 0·01N-BrCl, by the procedure previously outlined. The error of the determination is ±2%. B. VASZKO

3327. **A new test for chloropicrin.** H. Daecke and R. Kraul (Inst. org. Chem., Chem. Staatsinst., Hamburg). *Z. anal. Chem.*, 1961, **178** (6), 412–414 (in German).—Chloropicrin (trichloronitromethane) may be detected by reaction with a series of reagents, 10% KBr - 10% KCN (1:1) in 60% ethanol, pyridine, and aq. aniline hydrochloride (10%). These reagents can be used as a mixture or successively, in the proportions 2:1:1. In the presence of chloropicrin an orange colour is produced. The test may be applied to the detection of chloropicrin in air (0·3 to 0·4 μg per ml), either as a reaction in soln. or as a spot reaction on filter-paper. T. R. ANDREW

3328. **Determination of water in 1,1-dimethylhydrazine, diethylenetriamine and mixtures.** H. G. Streim, E. A. Boyce and J. R. Smith (Liquid Rocket Propulsion Lab., Picatinny Arsenal, Dover, N.J., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 85–89.—Two methods are described and compared. In one, the water is measured spectrophotometrically in the 1·7 to 2·2-μ range, a dry reference sample being prepared by passage through a column of Linde 4A Molecular Sieve. In the other, the water is determined by gas - liquid chromatography, with Theed (tetrahydroxyethylethylenediamine) as the stationary liquid phase supported on Chromosorb W. The precision of the first method is ±0·06%, and that of the second is ±0·1%. G. P. COOK

3329. **Determination of primary and secondary aliphatic amines by condensation with carbon disulphide.** N. Bellen and Z. Bellen (Struszyński Anal. Lab., Inst. of Gen. Chem., Warsaw). *Chem. Anal., Warsaw*, 1961, **6** (1), 63–67.—The following method, based on the work of Critchfield and Johnson (*Anal. Abstr.*, 1956, **3**, 2776), was developed for testing the condensate obtained in the industrial production of amines from alcohol and NH_3. Ammonia (>2 millimoles; 0·1 g as NH_4Cl) is removed from the sample as cobaltinitrite (*Anal. Abstr.*, 1961, **8**, 1473), the amines to be determined are converted into dithiocarbamic acids with CS_2, and the determination is completed by potentiometric titration. *Procedure*—To a sample containing ⩾20 milli-equiv. of amines and ⩾30 milli-equiv. of NH_3 add 5 ml of pptg. agent (12 g

of sodium cobaltinitrite, 12 g of $NaNO_2$ and 100 ml of water). Digest for 15 min., filter, and wash the ppt. with water and 30 ml of acetone. Treat the filtrate and washings with 30 ml of n-butanol and 5 ml of pyridine, then titrate potentiometrically with 0·5N-NaOH. Add 5 ml of CS_2, repeat the titration and complete by one of the usual methods.
P. BRYCH

3330. **Determination of tertiary amines in kerosine solutions in the presence of n-octanol.** C. Deptuła (Dept. of Chem. Technol., Inst. of Nuclear Res., Polish Acad. Sci., Warsaw). *Chem. Anal., Warsaw*, 1961, **6** (1), 91–94.—The method of Wagner *et al.* (*J. Amer. Chem. Soc.*, 1947, **69**, 2611), in which the primary and secondary amines are acetylated and the tri-n-octylamine is titrated potentiometrically with $HClO_4$ in the presence of glacial acetic acid, was suitable for this determination. Although kerosine and acetic anhydride are not miscible, the acetylation is quantitative and the presence of kerosine, mono- and di-octylamines and n-octanol does not affect the voltage break of the titration.
P. BRYCH

3331. **Determination of unsaturation in high-molecular-weight fatty nitrogen derivatives. Modification of the Wijs iodine-value method.** A. J. Milun (General Mills, Inc., Minneapolis, Minn., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 123–125.—In the application of the Wijs method to aliphatic nitrogen derivatives, primary and secondary amines were acetylated with acetic anhydride before adding the ICl and mercuric acetate reagents, tertiary amines were dissolved in acetic acid instead of $CHCl_3$, and quaternary ammonium chlorides were treated with sodium lauryl sulphate before the titration.
G. P. COOK

3332. **Thin-film ionophoresis and thin-film ionophoresis - chromatography [of amines and amino-acids].** C. G. Honegger (Forschungslab., Neurolog. Univ.-Poliklinik, Basel, Switzerland). *Helv. Chim. Acta*, 1961, **44** (1), 173–179 (in German).—The use of thin films of a solid adsorbent (Kieselgel G, Kieselgur G or alumina G), spread on a glass plate, for ionophoresis or combined ionophoresis and chromatography is described. Details are given of the apparatus.
G. RUSSELL

3333. **The use of acetic anhydride as a differentiating solvent for the selective acidimetric titration of amines, sulphoxides and amides.** N. N. Bezinger, G. D. Gal'pern and M. A. Abdurakhmanov (Inst. of Petrol. Chem. Synthesis, Acad. Sci. USSR, Moscow). *Zhur. Anal. Khim.*, 1961, **16** (1), 91–95.—It is shown that acetic anhydride is a differentiating solvent for organic bases and it is possible to titrate successively from a single sample in acetic anhydride aliphatic amines, aromatic (or heterocyclic) amines, sulphoxides and carboxylic acid amides. A sample containing from 0·0001 to 0·001 mole of each component is dissolved in 50 to 75 ml of acetic anhydride and titrated potentiometrically with 0·1N- or 0·05N-$HClO_4$, with glass and saturated calomel electrodes. If the sample is not easily dissolved, a small quantity of nitromethane, dioxan or chlorobenzene is added without interfering in the titration.
A. BURWOOD-SMITH

3334. **Application of chelatometry to the volumetric determination of diacetyl monoxime.** C. Hennart and E. Merlin (Orgafrance, Lab. de Villiers-St.-Georges, Seine-et-Marne, France). *Chim. Anal.*, 1961, **43** (1), 28–30.—A portion of the sample containing about 0·5 g of diacetyl monoxime is added to 25 ml of hydroxyammonium chloride soln. (0·5N) in the presence of 25 ml of Ni acetate soln. (0·2M). After being boiled for 4 hr. the soln. is cooled, 50 ml of aq. NH_3 soln. is added and the mixture is filtered and diluted to 250 ml. A 25-ml aliquot is titrated with EDTA (disodium salt) soln. (0·02M) to the purple end-point of murexide indicator.
G. P. COOK

3335. **Simple titrimetric method for the assay of thiols.** B. Saville (The British Rubber Res. Ass., 48–56 Tewin Road, Welwyn Garden City, Herts., England). *Analyst*, 1961, **86**, 29–32.—To 15 ml of pyridine are added an accurately weighed amount (0·0010 to 0·0018 mole) of the thiol sample and then gradually 5 ml of aq. $AgNO_3$ soln. (0·4M). After 5 min., water ($\simeq$100 ml) is added and the liquid is titrated with 0·1N-NaOH to phenolphthalein. The end-point may appear as a change in the colour of the yellow silver mercaptide to white, the pink colour of the indicator being almost complementary to yellow. With AnalaR pyridine, correction for a blank proved unnecessary. Each ml of 0·1N-NaOH $\equiv$ 0·0001 g of –SH. Quoted results show that the method can be applied to the assay of both saturated and unsaturated primary and secondary thiols and to tertiary aliphatic thiols.
A. O. JONES

3336. **Determination of mercaptans [thiols] by titration with mercury(II).** J. S. Fritz and T. A. Palmer (Iowa State Univ. Sci. and Technol., Ames, U.S.A.). *Anal. Chem.*, 1961, **33** (1), 98–100.—Samples ($\equiv$ 0·3 to 1 millimole of thiol) are titrated with $Hg(ClO_4)_2$ soln. in neutral aq. or acetone medium. The end-point is detected visually with Michler's thioketone as indicator or potentiometrically with a mercury indicator electrode. Smaller amounts of thiols in hydrocarbon soln. are titrated photometrically. The principal interfering substances are elementary S, S^{2-}, I^-, CN^- and thiocarbonyl compounds. The average error is $\pm$0·5%.
G. P. COOK

3337. **Gas-liquid chromatographic separation and polarographic reduction of thiosulphonates.** D. Barnard, M. B. Evans, G. M. C. Higgins and J. F. Smith (Nat. Rubber Producers' Res. Ass., Welwyn Garden City, Herts., England). *Chem. & Ind.*, 1961, (1), 20–21.—Suitable conditions are discussed for the analysis of thiosulphonates. Alkyl- and aryl-thiosulphonates give single-wave polarograms in soln. of pH $\simeq$1·0. The solvent used was benzene - water - methanol (1:2:7) and the electrolyte was an HCl - NH_4Cl buffer soln. Well-defined waves are given, the heights varying linearly with concentration up to 2×10^{-3}M, above which maxima are observed. The lower limit of detection is $\simeq$50 μg, and the polarographic method, while having an accuracy comparable to that of the titrimetric method (Barnard and Cole, *Anal. Chim. Acta*, 1959, **20**, 540), is not as sensitive as the colorimetric method *loc. cit.*). The mechanism of the polarographic reduction is discussed. Alkylthiosulphonates were separated chromatographically in Pyrex-glass U-tubes (5 ft. by 0·4 cm intern. diam.) packed with 5% of silicone fluid 704 (Hopkin & Williams) on 60 to 70-mesh Celite previously washed with 1% H_3PO_4 soln., equipped with a modified flame-ionisation detector. The columns were maintained at 138° by means of a n-pentanol vapour jacket. The separation of the lower alkylthiosulphonates is

shown by a comparison of the relative retention times in R_{x_9} units (Smith, *Chem. & Ind.*, 1960, 1024). R. A. HENDEY

3338. **Determination of benzenic (and aromatic) hydrocarbons in presence of terpenes in a mixed solvent fraction boiling below 200°.** G. Dupin. *Peint.-Pigm.-Vern.*, 1960, **36** (11), 638–641.—A scheme for the determination of aromatic hydrocarbons in (paint) solvent mixtures containing terpenes is outlined. Terpenes are removed by initial treatment with conc. H_2SO_4 and the benzenic and aromatic hydrocarbons determined from the absorption of HNO_3 (*d*, 1·49). L. A. O'NEILL

3339. **Polarographic determination of divinylbenzene in the products of the dehydrogenation of diethylbenzene.** J. Paściak (Res. Lab., Chem. Works, Oświęcim, Poland). *Chem. Anal., Warsaw*, 1961, **6** (1), 99–110.—The chief products of catalytic dehydrogenation of diethylbenzene are divinylbenzene (**I**) and ethylvinylbenzene (**II**), with a little naphthene. These were separated by chromatography on an alumina column, with 6% benzene in heptane as eluent. Polarograms obtained on successive fractions showed that **I** is subject to a two-step reduction, and **II** to a one-step reduction at the dropping-mercury electrode. The $E_{\frac{1}{2}}$ *vs.* the S.C.E. are −2·15 V and −2·48 V for the *p*-isomer of **I**, −2·26 V and −2·43 V for the *m*-isomer, and −2·45 V for a mixture of isomers of **II**. Proton donors, *e.g.*, methanol, ethanol or phenol, separate the waves sufficiently for a quantitative determination, as there is a linear relationship between the limiting current and the concn. of **I** in **II**. Styrene and naphthene do not interfere. Results obtained by this method were better than those given by the usual separation with N_2O_3. P. BRYCH

3340. **Counter-current distribution studies on phenols. II. Separation and estimation of lower phenols.** N. C. Saha and N. G. Basak (Central Fuel Res. Inst., Jealgora). *J. Sci. Ind. Res., India, D*, 1961, **20** (1), 11–15.—The separation of phenols between cyclohexane and aq. 10% NaCl by counter-current distribution in a 50-tube semi-automatic glass apparatus is described. Mixtures of phenol (**I**), *o*-cresol (**II**), *m*-cresol (**III**) and *p*-cresol (**IV**) were used, and the upper cyclohexane layer was analysed in each tube by measuring the extinction at 279 mμ. It was found that **I** was present in tubes 4 to 22, with a max. in tube 12; **II** was present in tubes 24 to 44, with a max. in tube 35; and, from extrapolated values, **III** was present in tubes 16 to 38, with a max. in tube 28. An equation is given for the calculation of each component using the sum of the extinctions of each tube in the distribution band. Mixtures of **III** and **IV** have a similar distribution and must be further separated by partition chromatography or by the extinction method of Padhye and Sule (*J. Sci. Ind. Res., India, B*, 1957, **16**, 326). Higher phenols, *e.g.*, ethylphenols and xylenols, have higher partition coefficients than the cresols and are therefore easily separated. Errors are <3%. R. A. HENDEY

3341. **Oscillopolarographic determination of hydroxybiphenyls in phenol production.** J. Drahoňovský and V. Racek (Spolana, Neratovice, Czechoslovakia). *Chem. Průmysl*, 1961, **11** (1), 22–24.—A mixture of thiophenol (**I**) and phenylphenols (**II**) in a soln. of N-NaOH is characterised by two incisions on the $dV/dt = f(V)$ curves; the first corresponds to **I**, the second to *o*- or *p*-phenylphenol. The differentiation of the mixture can be made when the ratio of **II** to **I** is in the range 10 : 1 to 1 : 5. The depth of the incisions can be compared with a calibration curve and used for the determination of 1·5 to 2·5 mg of **I** in 100 ml of sample. Biphenyl oxide, biphenyl sulphone, Na benzenesulphonate and resorcinol do not interfere. By deducting the amount of **I** found oscillographically from the values found by bromination of the sample, the amount of phenol in the sample can be determined. Errors caused by the presence of **I** do not influence the results. J. ZÝKA

3342. **Detection of *p*-cresol in spot-test analysis.** F. Feigl and V. Anger (Min. Agric., Rio de Janeiro, Brazil). *Anal. Chem.*, 1961, **33** (1), 89–90.—When coupled with diazotised *p*-nitroaniline and then treated with MgO in alkaline medium, *p*-cresol produces a blue lake. The other isomers and phenol do not interfere; 0·2 μg can be detected. G. P. COOK

3343. **Indirect complexometric determination of catechol.** Z. Łada and J. Młodecka (Struszyński Anal. Lab., Inst. of Gen. Chem., Warsaw). *Chem. Anal., Warsaw*, 1961, **6** (1), 95–98.—*Procedure*—Dissolve the sample containing 0·03 to 0·06 mg of catechol in ≃50 ml of water at 60° to 70° and add 20 ml of 10% Pb acetate soln. Cool the mixture for 30 min., then filter through a G4 sinter. Wash the ppt. with cold CO_2-free water (6 × 15 ml), dissolve it in 20 ml of 2N-HNO_3, and make the soln. up to 100 ml. Make a 25-ml aliquot slightly ammoniacal, add 10 ml of buffer soln. (NH_4Cl - aq. NH_3) (pH 10) and 20 ml of 0·01M-EDTA (disodium salt). Titrate the excess of EDTA with 0·01M-$MgCl_2$, with Eriochrome black T as indicator; 1 ml of EDTA (0·01M) ≡ 1·1011 mg of catechol. Resorcinol and quinol do not interfere. The method is evaluated statistically and compared with gravimetric and manganometric methods. Attempts to determine residual Pb after pptn. gave scattered and inaccurate results owing to the large excess of Pb required for complete pptn. P. BRYCH

3344. **Gas-chromatographic analysis of chloronitrobenzene isomers.** K. J. Bombaugh (Res. and Dev. Div., Spencer Chemical Co., Merriam, Kans., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 29–32.—Complete separation has been achieved on a commercial instrument by the use of a 4-metre column of 60 to 80-mesh Chromosorb washed with aqua regia and loaded with 4M Carbowax 1000, operating at 152° with a flow rate of 46·2 ml per min. E. G. CUMMINS

3345. **Infra-red spectroscopic method for the determination of residues of pentachloronitrobenzene.** G. Paulig. *Dtsch. LebensmittRdsch.*, 1960, **56** (10), 296–298.—The method is based on reduction of pentachloronitrobenzene to pentachloroaniline, in 98% yield, with zinc and acetic acid, and measurement of its infra-red absorption in the 7·23-μ band, with CCl_4 as solvent. *Procedure*—Vigorously stir the shredded sample (*e.g.*, cabbage) (200 g) for 30 min. with CCl_4 (300 ml), filter the extract through a G4 sintered glass filter and wash the residue with CCl_4 (300 ml). Reduce the vol. of the filtrate to 10 ml, dry with Na_2SO_4 and transfer to a column of activated alumina (10 cm). Elute with CCl_4 (150 ml) and evaporate to dryness. Add zinc (3 g) and 75% acetic acid (100 ml) and boil under reflux for 1 hr. Dilute threefold with water and

extract with CCl_4 (3 × 50 ml). Wash the combined extracts with water, dry with Na_2SO_4 and evaporate to dryness under vacuum. Boil the residue for 30 min. with 2N-ethanolic KOH (250 ml), dilute with water and extract with CCl_4 (2 × 50 ml). Evaporate under vacuum, and to the residue add water (500 ml) and steam-distil, collecting about 300 ml. Extract the distillate with CCl_4 (2 × 50 ml), dry and evaporate. Dissolve the residue in CCl_4 (5 ml) and measure the i.r. absorption with a 5-mm cell. By this method 0·1 p.p.m. of pentachloronitrobenzene in cabbage was determined with a max. error of ≃15%. E. C. APLING

3346. **Micro-determination of *p*-aminophenol.** E. Jungreis and J. C. Soriano (Dept. of Inorg. and Anal. Chem., Hebrew Univ., Jerusalem). *Mikrochim. Acta*, 1961, (1), 26–31 (in English).—Mild aerial oxidation of *p*-aminophenol (**I**) followed by reaction with phenol gives a blue colour. Excess of *m*-aminophenol or other aromatic amines can be tolerated, but *o*-aminophenol interferes slightly. *Procedure*—Adjust 1 ml of ethanolic **I** soln. (≮0·01 mg) to pH 10 with 2 ml of buffer soln. (100 ml of 0·05M-$Na_2B_4O_7$ and 6·7 ml of 0·1M-NaOH), add 1 ml of 10% aq. phenol soln. and dilute to 10 ml. After 2 hr. measure the extinction at 620 mμ. In the presence of *m*-aminophenol, use this compound in place of phenol and measure at 570 mμ.
P. D. PARR-RICHARD

3347. **Determination of 2,4-diaminophenol and its salts.** D. V. Parke (Biochem. Dept., St. Mary's Hospital Med. Sch., London). *Analyst*, 1961, **86**, 12–15.—To 2 ml of a soln. of 2,4-diaminophenol dihydrochloride (Amidol) in 0·5N-HCl (10 to 250 mg per ml) are added 1 ml of $K_2S_2O_5$ soln. (0·2% w/v) and 2 ml of M-Na acetate. After 10 min. the liquid is diluted and its extinction is measured against water at 500 mμ. Beer's law is obeyed over the range 0 to 250 μg per ml and the method is sensitive to 10 μg. Other related amines and phenols do not interfere. 2,4-Diaminophenol is excreted as a metabolite of 2,4-dinitrophenol and other compounds and occurs in the urine as conjugates of acetic, sulphuric and glucuronic acids, which are hydrolysed with HCl. The method can be applied to urine by boiling the sample under reflux with 5N-HCl for 3 hr. The recovery from rabbit urine was 101 ± 3%. In the presence of *m*-phenylenediamine, *m*-nitroaniline, 2-amino-4-nitrophenol or D-glucuronolactone (100 mg of each), recoveries were 111, 90, 93 and 102%, respectively. Corrections should be made for the presence of large amounts of these substances. A. O. JONES

3348. **Argentimetric determination of 2-mercaptobenzothiazole in technical and crude products.** W. Czerwiński and H. Vieweger (Anal. Dept., Inst. Gen. Chem., Warsaw, Poland). *Chem. Anal., Warsaw*, 1960, **5** (6), 1011–1019.—Two potentiometric methods for the determination of 2-mercaptobenzothiazole (**I**) are described. When sym.-diphenylthiourea (**II**) is absent, the potentiometric titration is carried out with silver - silver chloride and mercury sulphate electrodes, with 0·1N-$AgNO_3$ as the titrant. When **II** is present as an impurity, silver - silver chloride and glass electrodes are used. The sample is dissolved in acetone and titrated with 0·1N-ethanolic $AgNO_3$ and 0·1N-ethanolic NH_3. These soln. are added alternately in 0·05-ml increments, starting with the $AgNO_3$ soln. The accuracy for **I** is ±0·27% by the first method, and ±0·55% by the second. B. K.

3349. **Detection of tetraphenylborate ions.** R. Neu (Firma Dr. Willmar Schwabe G.m.b.H., Karlsruhe, Germany). *Mikrochim. Acta*, 1961, (1), 32–36 (in German).—The reaction of tetraphenylborate with 8-hydroxyquinoline can give two different products according to the method of preparation. One of these compounds, which has been prepared previously (Mukherji and Sant, *Ibid.*, 1959, 370), is compared with that obtained by the author. The reactions are useful for preparing derivatives of suitable hydroxyarylamines or hydroxyhetero(*N*)hydrocarbons.
P. D. PARR-RICHARD

3350. **Fluorescence analyses for polycyclic aromatic hydrocarbons.** J. H. Chaudet and W. I. Kaye (Tennessee Eastman Co., Kingsport, U.S.A.). *Anal. Chem.*, 1961, **33** (1), 113–117.—Concentration quenching, localisation of fluorescence and self-absorption effects can be minimised if an optimum solute concn. is used. This optimum concn. for several polycyclic hydrocarbons has been determined from their absorption of the excitation energy at 365 mμ. G. P. COOK

3351. **Chromatography of terpene hydrocarbons.** I. Gallardo and A. L. Montes (Univ. of Buenos Aires, Argentina). *An. Asoc. Quim. Argentina*, 1960, **48** (2), 108–116.—The analysis of mixtures of terpene hydrocarbons has been effected by displacement chromatography on a silica-gel column. The mixture is freed from oxygenated compounds, dissolved in a paraffinic hydrocarbon, applied to the column and displaced with benzene or toluene. Examples of the separation of synthetic mixtures, turpentine and the oil from the oleoresin of the California pepper tree (*Schinus polygamus*) are given. L. A. O'NEILL

3352. **Identification of pyrimidine derivatives on a micro scale by complex formation.** R. Weiss (Inst. für Mikrobiol. u. exp. Therapie, Jena, Germany). *Mikrochim. Acta*, 1961, (1), 11–25 (in German).—Purine and pyrimidine derivatives are difficult to identify by m.p. because of decomposition. Identification of ≃100 compounds by solubility tests and colour reactions is described. If the sample (0·5 to 1 mg) is insol. in N-NaOH, its solubility in acetone, $CHCl_3$ and CCl_4 is used to assign it to a narrow group. Other compounds, sol. in NaOH, are identified by colour reactions with NaOH, HNO_3, $AgNO_3$, $CuSO_4$, $CoSO_4$, $Co(SCN)_2$, Fe^{3+}, picric acid and $Ba(OH)_2$. Light absorption and fluorescence provide further confirmation. P. D. PARR-RICHARD

3353. **Quantitative determination of free sulphur in hydrocarbons by the benzoin method.** A. B. Stern and M. Babitz (Chem. Testing Lab., Technion-Israel Inst. Technol., Haifa). *Bull. Res. Council Israel, C*, 1960, **8** (3), 109–116.—The free S is converted into H_2S by reaction with benzoin at 180° and the H_2S is determined by the methylene blue method of Jacobs *et al.* (*Anal. Chem.*, 1957, **29**, 1349). Combined S, except in a few olefinic sulphur compounds, has no effect. The range of detection is 0·5 to 100 μg per ml, and the accuracy varies from 70 to 100%. S.C.I. ABSTR.

3354. **Determination of sulphur in gasoline by X-ray emission spectrography.** R. A. Jones (Ethyl Corp. Res. Lab., Detroit, Mich., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 71–74.—Matrix effects are avoided by diluting one portion of the sample with

sulphur-free iso-octane, while an equal amount of diluent containing a known amount of sulphur is added to another portion. The sulphur content of the sample is then determined from the intensities of the sulphur K_α radiation and the background radiation of the two soln. Concn. down to 0·002% can be determined, and the estimated standard deviation is ±0·00115% absolute. G. P. Cook

3355. **Spectrophotometric determination of manganese in gasoline.** E. D. Steinke, R. A. Jones and M. Brandt (Ethyl Corp., Detroit, Mich., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 101–102.—The manganese compound present, namely (methylcyclopentadienyl)manganese tricarbonyl, is decomposed with a soln. of Br in CCl_4. The Mn is extracted into H_3PO_4, the organic material in the extract is oxidised with HNO_3 - H_3PO_4, and further oxidation with KIO_3 gives permanganate, the extinction of which is measured at 525 mμ. Results are accurate to within ±2%. G. P. Cook

3356. **Direct nickel determinations in petroleum oils by X-ray at the 0·1-p.p.m. level.** C. C. Hale and W. H. King, jun. (Esso Res. & Engng Co., Linden, N.J., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 74–77.—The electronics and optics of X-ray fluorescence instrumentation, together with the technology and a matrix compensation method, were improved to lower the limit of detection of Ni to less than 0·1 p.p.m. In the range from 0 to 0·6 p.p.m. the standard deviation was ±0·035 p.p.m. G. P. Cook

3357. **Determination of organic salts [in lubricating oils] by conductimetry in a benzene - ethanol medium.** J. Larbre and J. Briant (Inst. Franç. Pétrole, Rueil-Malmaison, S.-et-O.). *Rev. Inst. Franç. Pétrole*, 1960, **15** (7–8), 1170–1180.—Organic salts such as the sulphonates and phenoxides of Ca and Ba, which are used as detergent additives in lubricating oils, can be determined precisely in low concn. in small samples by titration in a mixture of benzene and ethanol (1:1 by wt.) with a soln. (in water, ethanol or benzene - ethanol) of a strong acid or of one which yields an insol. product. The choice of titrant is discussed. It is possible to determine the basicity of a salt and to distinguish between Ca and Ba. No preliminary treatment of the sample is necessary. G. Burger

3358. **Detection of glycols in crank-case oils.** C. B. Jordan (Aberdeen Proving Ground, Md., U.S.A.). *Mater. Res. & Stand.*, 1961, **1** (1), 30.—The vicinal glycol is recovered by azeotropic reflux distillation of the oil (100 ml) with xylene in a modified Dean and Stark apparatus, and is then identified by adding 5 ml of the aq. layer to a mixture of 0·04N-HIO_4 (5 ml), conc. HNO_3 (4 drops) and 0·1N-$AgNO_3$ (3 ml). The formation, after thorough shaking, of a dense white ppt. of $AgIO_3$ within 30 sec. indicates the presence of glycol in concn. as low as 1%. W. J. Baker

3359. **Determination of organically combined sulphur in town gas and similar gases by a new combustion method.** K. Günther (Zentrallab., Berliner Gaswerke, Berlin). *BrennstChemie*, 1960, **41** (11), 326–329.—A procedure is described for the determination of as little as 0·005 millimole of S per litre of gas. The gas is burned in air and the products are absorbed in 3% H_2O_2 soln. The H_2SO_4 produced is titrated conductimetrically with NaOH. The method is rapid and precise. The apparatus comprises a quartz tube fitted with a small heater (800°), a burner nozzle with a purified air supply and an absorption tube. The gas sample can be metered into the apparatus directly at the sampling point or collected in a gas burette. G. Burger

3360. **Analysis and characterisation of coal tars by solvent extraction and chromatography.** G. V. Blunt and M. Vahrman (Northampton Coll. of Adv. Technol., London, E.C.1). *J. Inst. Fuel*, 1960, **33**, 522–529.—The tar is extracted successively with benzene and pyridine, and the benzene-sol. fraction with light petroleum, and the basic and acidic portions are extracted from the pyridine-sol., light petroleum-sol. and light petroleum-insol. fractions with 2N-H_2SO_4 and 2N-KOH, respectively, then liberated with 2N-KOH and 2N-H_2SO_4, respectively, and extracted with $CHCl_3$. The neutral residues are chromatographed on a silica-gel column in light petroleum; four fractions are obtained on elution with, respectively, light petroleum (fluorescent), light petroleum (non-fluorescent), benzene and ethanol. The procedure can be adapted to high-temp. tars and to those containing volatile constituents. The losses at various stages of the analysis are discussed. D. De Voy

3361. **Direct determination of organic oxygen in coals.** A. Crawford, M. Glover and J. H. Wood (Nat. Coal Board, N.W. Div., Central Lab., Wigan, Lancs.). *Mikrochim. Acta*, 1961, (1), 46–58 (in English).—Modifications to the Unterzaucher method (*Analyst*, 1952, **77**, 584), which is based on high-temp. reduction of O by carbon, are described. After extraction of interfering mineral matter with HCl, the sample is pyrolysed in N at 900° in contact with platinised carbon (50% of platinum), the CO produced is oxidised with I_2O_5 and the liberated iodine is titrated. Full experimental details are given. P. D. Parr-Richard

3362. **Determination of arsenic in malting coal.** R. G. Ault (Mitchells & Butlers Ltd., Cape Hill Brewery, Birmingham, England). *J. Inst. Brewing*, 1961, **67** (1), 14–28.—An account is given of collaborative studies made by the Institute of Brewing and by the National Coal Board which have led to the acceptance by the B.S.I. of two methods of analysis. These have in common a procedure for the destruction of organic matter, which involves igniting the sample, in admixture with MgO and $KMnO_4$, in a stream of oxygen. This is followed, in one method, by a colorimetric molybdenum blue procedure, as described by Crook and Wald (*Anal. Abstr.*, 1961, **8**, 1566), and, in the other, by the Gutzeit method as described by Ault and Whitehouse (*Ibid.*, 1957, **4**, 3914). Within- and between-laboratory tolerances were both 1 p.p.m. of As_2O_3 for samples containing <6 p.p.m., and 1·5 p.p.m. for samples containing >6 p.p.m., when the colorimetric procedure was employed. The corresponding tolerances for the Gutzeit method were 1·5 and 2·5 p.p.m., respectively. Differences between values obtained by the two methods were negligible in comparison with errors that might arise from difficulties of sampling. J. R. Hudson

3363. **[Determination of] the amide nitrogen content of gelatins.** J. E. Eastoe, J. E. Long and A. L. D. Willan (Brit. Gelatine and Glue Res. Ass., 2A Dalmeny Avenue, London, England). *Biochem. J.*, 1961, **78** (1), 51–56.—A rapid and convenient

method is described. It is based on a preliminary hydrolysis with 2N-HCl at the b.p. for 1 hr. The cold hydrolysate is neutralised to a pH of ≃5·2 with 2N-NaOH and then steam-distilled in the presence of 0·15N-NaOH and 0·05M-$Na_2B_4O_7$. The distillate is collected in 1% aq. H_3BO_3 and this soln. is titrated with 0·01N-HCl. Under these conditions there is no significant decomposition, resulting in release of non-amide NH_3, during the distillation stage. The method of hydrolysis with 2N-HCl at the b.p. gives results that are very close to those obtained after hydrolysis with 12N-HCl at 37°. J. N. ASHLEY

3364. **Determination of biologically soft and hard alkylbenzenesulphonates in detergents and sewage.** C. P. Ogden, H. L. Webster and J. Halliday (Thomas Hedley & Co. Ltd., Newcastle-upon-Tyne, England). *Analyst*, 1961, **86**, 22–29.—The sample (≡≃1 g of alkylbenzenesulphonate) is dissolved in 1 litre of water and a 100-ml aliquot is boiled with 25 ml of conc. HCl. The liquid, re-adjusted to 100 ml, is extracted with light petroleum under specified conditions, the extract being finally discarded. The aq. residue, adjusted to a known ethanol - water ratio (1:4), is treated with heptylamine and the heptylammonium salt is extracted with light petroleum. The residue from the evaporated, filtered extract is dissolved in methanol (50 ml) and a 5-ml and a 25-ml portion are evaporated separately. The residue from evaporation of the 5-ml portion is dissolved in CS_2 and its i.r. extinction is measured at 9·9 μ and referred to a calibration graph to obtain the content of total alkylbenzenesulphonate. The residue from the 25-ml portion is dissolved in CCl_4 and its i.r. extinction is measured at 7·31 μ to obtain the content of hard (*i.e.*, branched-chain) alkylbenzenesulphonate. The soft (*i.e.*, straight-chain) alkylbenzenesulphonate content is obtained by difference. For sewage the detergent is adsorbed on carbon and eluted with methanol - $CHCl_3$ - aq. NH_3, and the method is applied to the residue after removal of the solvent. A. O. JONES

3365. **Determination of surface-active chelating agents.** S. Mook, E. Schonfeld and D. Ghosh (Radiation Applications Inc., Long Island City, N.Y., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 150–151. —The method of Darbey (*Ibid.*, 1952, **24**, 373) was adapted to the determination of dodecylbenzyldiethylenetriaminetetra-acetic acid, which is used for the foam separation of metals in nuclear processes. G. P. COOK

3366. **Detection and estimation of polyoxyethylene glycol in non-ionic surfactants by ascending paper chromatography.** M. E. Ginn, C. L. Church, jun., and J. C. Harris (Monsanto Chemical Co., Dayton, Ohio, U.S.A.). *Anal. Chem.*, 1961, **33** (1), 143–145. —Samples are applied to the base of a paper cylinder and the chromatogram is developed with n-butanol saturated with water and acetic acid. The spots are detected with a modified Dragendorff reagent. The polyoxyethylene glycol is determined from the areas of the spots. Recoveries on samples containing 4 to 15% of the glycol are 94 to 98%. G. P. COOK

3367. **Determination of carbonyl groups in cellulose by sodium borohydride reduction.** A. Sugimatsu and S. Yahara. *J. Jap. Tech. Ass. Pulp Paper Ind.*, 1960, **14** (8), 516–521 (in Japanese, English summary).—The volumetric method of Lyttle *et al.* (*Anal. Chem.*, 1952, **24**, 1843) was preferred to the gasometric method of Lindberg and Misiorny (*Bull. Inst. Pap. Chem.*, 1952, **22**, 485) and modified. The material to be analysed (*e.g.*, D-glucose or periodate oxycellulose) is immersed in 0·01M to 0·13M-$NaBH_4$ (pH 12·0 to 12·3) for 24 to 48 hr. at room temp. The soln. is then filtered, and the filtrate is treated successively with 0·04N to 0·4N-KIO_3 and 0·5N-NaOH. The amount of $NaBH_4$ consumed is calculated from the iodate consumption as established by titration with 0·1N-$Na_2S_2O_3$ under acid conditions. In the pH range of 12·0 to 12·3, 57% of the $NaBH_4$ consumed is effective in reducing the carbonyl groups of the test material. Carbonyl determinations on oxycellulose and D-glucuronolactone showed that carboxyl and lactone groups are resistant to borohydride reduction. ABSTR. BULL. INST. PAPER CHEM.

3368. **Method for the quantitative chemical analysis of mixtures of cellulose triacetate and secondary cellulose acetate fibres.** British Standards Institution (2 Park Street, London, W.1). B.S. 3334:1961. 9 pp.—The secondary cellulose acetate is removed by dissolution in acetone. The residue is collected, washed, dried and weighed and, after corrections have been applied, is expressed as cellulose triacetate. The proportion of secondary cellulose acetate is found by difference.

3369. **Colorimetric method for the determination of acetic acid in wood hydrolysate containing a large amount of sulphuric acid.** Yoshio Sakai and Wasako Kumagai (Fac. of Agric., Tokyo Univ. of Education). *Agric. Biol. Chem., Japan*, 1961, **25** (1), 71–73.—The reaction between aniline, furfuraldehyde and acetic and sulphuric acids in butanol soln. yields a coloured product which may be utilised for the determination of acetic acid. An excess of $BaCO_3$ is added to the sample and the ppt. is filtered off. The combined filtrate and washings are diluted so that the concn. of acetate will be 0·2 to 1 mg per ml and an aliquot (5 ml) is acidified with 0·2N-H_2SO_4 (1 ml) and extracted with n-butanol (15 ml); 10 ml of the butanol layer is treated with 1% ethanolic aniline soln. (2 ml) and 1% ethanolic furfuraldehyde soln. (2 ml). Colour due to H_2SO_4 is eliminated by treatment with $BaCO_3$, and the extinction at 510 mμ is measured. The colour is decreased by rise in temp., and acids that give sol. barium salts interfere. J. R. HUDSON

3370. **Determination of non-cotton content of bleached cotton textiles. Tentative Test Method 97–1960.** Amer. Ass. Text. Chemists and Colorists Committee on Bleaching. *Amer. Dyest. Rep.*, 1960, **49** (17), P606.—Water-soluble and enzyme-extractable materials are determined in the dried sample by weighing before and after successive treatments with hot water and bacterial amylase soln. Oils, fats and waxes are then removed from the dry residue by extraction with trichloroethylene and the final residue is ignited at 600° to 650° to determine ash. E. S. LANE

3371. **The quantitative analysis of binary fibre mixtures containing Courtelle (acrylic fibre).** W. Armfield (Courtaulds Ltd., Droylsden, Manchester). *J. Text. Inst. Proc.*, 1960, **51** (12), P699–P705.—Courtelle is dissolved from its mixture with silk, cotton, wool, nylon 6·6, Terylene, Fibro (viscose rayon), or other acrylic fibres (except basified

acrylics dyed with chromed acid dyes) with dimethylformamide at 60°, or, in special cases, at 90°. Detailed procedures are given.

O. M. WHITTON

3372. **Quantitative chemical analysis of ternary mixtures of protein fibres, polyamides and certain other fibres.** British Standards Institution (2 Park Street, London, W.1). B.S. 3336:1961. 8 pp. —The methods are based on those described in B.S. 2822:1957 and B.S. 3069:1959. It is assumed that the fibres present have been identified previously.

3373. **Spectrophotometric determination of hydroxyl groups in poly[oxy]propylene glycols.** D. Kyriacou (Aerojet-General Corp., Sacramento, Calif., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 153–154. —The method is based on the direct spectrophotometric titration (at 2·87 to 2·88 μ) of the hydroxyl compound with a standard solution of acetyl chloride. Correction for water content is made after its determination by the Karl Fischer method.

G. P. COOK

3374. **Paper-chromatographic separation and identification of poly[oxy]ethylene glycols and their monoethers.** J. Borecký and J. Gasparič (Mikroanal. Lab., Forschungsinst. für org. Synthesen, Pardubice-Rybitví, Czechoslovakia). *Mikrochim. Acta*, 1961, (1), 96–104 (in German).—The alcohols are converted into 3,5-dinitrobenzoates by the reaction of 0·05 ml with a little 3,5-dinitrobenzoyl chloride for 10 to 15 min. at 100° to 110°. Five drops of 5% Na_2CO_3 soln. and 1 ml of benzene are added to the cooled soln., which is then transferred to a paper impregnated with formamide or dimethylformamide (**I**). The chromatogram is developed with suitable mixtures of hexane, cyclohexane, benzene and $CHCl_3$, or a paper impregnated with liquid paraffin can be used with **I** - methanol - H_2O (4:1:1) as the mobile phase. The spots, after reduction with $ZnCl_2$, are identified with *p*-dimethylaminobenzaldehyde soln. Tables of R_F values are given.

P. D. PARR-RICHARD

3375. **Liquid chromatographic separation of mixtures of polymers and mineral oil.** P. I. Brewer (Esso Res. Ltd., Abingdon, Berks., England). *Nature*, 1960, **188**, 934–935.—Polymers dissolved in mineral oil can be separated by a technique similar to that for macromolecules in aq. soln. (*cf.* Porath and Flodin, *Anal. Abstr.*, 1960, **7**, 803). The column (21 mm in diam. by 82 cm in length) was packed tightly with small pieces of thin vulcanised natural rubber latex. The rubber was washed with n-heptane before being placed in the column. The sample (≃250 mg) was dissolved in 5 ml of n-heptane and placed on top of the rubber packing. When all the sample had passed into the packing, further solvent was added, and the flow-rate adjusted to between 10 and 15 ml an hour. Fractions of 6 ml were collected, then evaporated and the residue weighed. A graph given shows good separation of polyisobutene (mean mol. wt. = 18,000) (**I**) and mineral oil, with a recovery of 98·2% of **I**. The rubber is slightly soluble in n-heptane (≃0·2 mg per 6-ml fraction). It is suggested that the separation is due to the swollen rubber acting as a type of molecular sieve, the small mineral-oil molecules diffusing into the rubber and so being delayed in their passage through the column. Other types of polymers in mineral oils have been separated by the method.

R. A. HENDEY

3376. **Rapid identification of high polymers using a simple pyrolysis unit with a gas - liquid chromatograph.** C. E. R. Jones and A. F. Moyles (Res. Dept., Vinyl Products Ltd., Carshalton, Surrey, England). *Nature*, 1961, **189**, 222–223.—The unit described is simple to construct and robust in operation. Characteristic finger-print chromatograms are shown for homopolymers of methyl methacrylate and styrene, and copolymers of methyl methacrylate with styrene and methyl methacrylate with α-methylstyrene.

C. B. BAINES

3377. **Estimating thermal stability of experimental polymers by empirical thermogravimetric analysis.** C. D. Doyle (Gen. Engng Lab., General Electric Co., Schenectady, N.Y., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 77–79.—Measurement of thermogram areas, to give what is defined as an integral procedural decomposition temperature (ipdt) has been used to provide reproducible data in terms of the refractory character and the stability of polymers. Applicable to cases of multi-stage decomposition, the ipdt is more appropriate than the more usual half-volatilisation temperature. Empirical values of ipdt reported range from 345° to 555° for Plexiglas and Teflon, respectively.

E. G. CUMMINS

3378. **An accurate colorimetric method for the determination of end-groups.** S. R. Palit (Indian Assoc. for Cultivn. of Science, Calcutta, India). *Kunststoffe*, 1960, **50** (9), 513–514.—A sensitive qual. test for higher polymers with ionising end-groups is described. *Procedure*—A small amount of polymer is dissolved in a few ml of $CHCl_3$, a few ml of methylene blue soln. (≃10 mg of dyestuff per litre of 0·01N-HCl) is added, and the mixture is shaken. When the polymer has a strongly acid end-group, the $CHCl_3$ layer is coloured immediately. The intensity of the colour depends on the proportion of ionising groups in the molecule. The test has been applied to polymethacrylates and polystyrene produced by various activation methods. The results are discussed. The test can be modified to detect other groups by changing the dyestuff and solvent. Polymers lacking ionising end-groups react to the test after a preliminary treatment.

A. C. R. HARTLEY

3379. **Detection of end-groups in poly(vinyl cyanide) by differential infra-red spectroscopy.** Ikuo Matsubara (Central Res. Lab., Toyo Rayon Co. Ltd., Otsu, Shiga). *Bull. Chem. Soc. Japan*, 1960, **33** (11), 1624–1626.—The end-groups in poly(vinyl cyanide) are determined by differential infra-red spectroscopy with the KBr disc technique. The limit of detectability of both sulphate and sulphonate end-groups by this method is estimated to be ≃0·002 millimole per g of polymer.

I. JONES

3380. **Determination of insoluble residue in vinyl cyanide - methyl methacrylate copolymer.** J. Majewska and S. Urbanowicz (Inst. of Artificial and Synthetic Fibres, Łódź, Poland). *Chem. Anal., Warsaw*, 1960, **5** (6), 1045–1048.—Poly(vinyl cyanide) (**I**) and poly(methyl methacrylate) (**II**) are present as impurities in the synthesis of vinyl cyanide - methyl methacrylate copolymer (**III**). It was found that **I** is insoluble in acetone, but **II** is quant. dissolved. The sample of **III** was extracted with acetone in a Soxhlet apparatus for 2·5 hr., dried and weighed, the loss in weight corresponding to the content of **II**. The standard deviation was 0·05 (10 determinations).

B. K.

3381. **Method for determination of nitrogen in vinyl cyanide polymers.** J. Majewska and M. Jordan (Inst. of Artificial and Synthetic Fibres, Łódź, Poland). *Chem. Anal., Warsaw*, 1960, **5** (6), 1039–1044.—*Procedure*—Place 0·06 to 0·08 g of sample in a Kjeldahl flask, add conc. H_2SO_4 (8 ml) and catalyst, and mineralise. Transfer an aliquot of the soln. to a Markham apparatus (*Biochem. J.*, 1942, **36**, 790) and distil the NH_3 into a flask containing satd. H_3BO_3 soln. (10 ml), H_2O (10 ml) and 2 drops of Tashiro indicator. Titrate the distillate with 0·02N-HCl. Five catalysts were tested, the best results being obtained with a mixture of $CuSO_4.5H_2O$ (0·31 g), mercury (0·14 g), $Na_2SO_4.10H_2O$ (1·8 g) and HgO (0·14 g). The mean result for N in poly(vinyl cyanide) was 26·27 ± 0·047% (20 determinations). B. K.

3382. **Quantitative determination of carboxyl groups in polyamides.** E. Ya. Valman, L. I. Pokrovskii and S. S. Frolov (Ivanov Chem.-Tech. Inst.). *Zhur. Prikl. Khim.*, 1961, **34** (1), 232–233.—*Procedure*—Dissolve 3 g of polyamide resin in 12 ml of phenol (85 to 90% aq. soln.) and mix until a homogeneous soln. is obtained, then precipitate the polyamide with 40 ml of ethanol. Dilute the suspension with 250 ml of H_2O, stirring slowly, then filter and wash the ppt. with warm water to remove phenol and ethanol. Transfer the ppt. (or degreased fibre) to a 200-ml flask and add 35% formaldehyde soln. (50 ml), previously neutralised with 0·1N-NaOH. Mix carefully and allow to stand, 45 to 60 min. for pptd. nylon or 75 to 90 min. for amide or nylon fibre. Then add 0·025N-NaOH (25 ml), set aside for 15 min. and make up the vol. with H_2O. Filter and titrate 50 ml of filtrate with 0·05N-HCl to the colour discharge of phenolphthalein. The mol. wt. of the polyamide is calculated as the reciprocal of the amount of carboxyl group (moles per g of polyamide).
A. Burwood-Smith

3383. **Determination of water in plastic materials.** V. W. Reid and L. Turner (Petrochemicals Ltd., Carrington Res. Lab., Urmston, Manchester, England). *Analyst*, 1961, **86**, 36–39.—The method is based on a Karl Fischer titration. The analytical train described consists of a pressure-control arm for the supply of N which passes through a drying tower [$Mg(ClO_4)_2$] into the sample contained in a glass tube immersed in an oil bath at 120°. The N stream then passes into a Karl Fischer titration apparatus designed for gases. A "dead-stop" end-point indicator consists of a potentiometer circuit applying an e.m.f. of 100 mV to the electrodes of the titration cell with a milliammeter in series. The Karl Fischer titrant is added from a microburette in 0·1-ml portions until the end-point indicator shows that the N stream is free from water. The results obtained indicate the reproducibility of the method to be ≃ ±10% of the water content when this is in the region of 0·0 to 0·5%. The method has proved particularly useful for the determination of water in black polyethylene.
A. O. Jones

3384. **Methods of evaluating the quality of plastic lubricants.** V. V. Sinitsyn. *Zavod. Lab.*, 1961, **27** (1), 47–58.—A review, with 136 references, is presented. G. S. Smith

3385. **Spectrographic examination of small samples by a procedure giving high sensitivity.** W. A. Cregeen (Attorney-Gen. Lab., Toronto, Canada). *J. Forensic Sci.*, 1960, **5**, 226–235.—A cathode-layer excitation method is described, applicable to the examination of paints and related material when duplicate spectrographic samples are limited to 2 mg each. A specially cut carbon cathode, 1·3 mm in diam., serves as the sample electrode and the method depends upon the projection of the image of the cathode, in the close vicinity of which there exists an enriched zone of ions and atoms due to cathodic migration. Data for 43 elements are presented; results are compared with those by a normal excitation method. A tenfold increase in absolute sensitivity was obtained. Chem. Abstr.

3386. **Polarographic determination of tetraethylthiuram disulphide in chloroprene latex.** J. Kresta and O. Mikl (Res. Inst. Synth. Rubber, Gottwaldov, Czechoslovakia). *Chem. Průmysl*, 1961, **11** (1), 52–55.—*Procedure*—To the supporting electrolyte (0·2M-LiCl, 0·025M-Na acetate, 95% methanol and 5% of H_2O) (50 ml) add dropwise, and with stirring, the sample of latex (1 ml), then set aside for 30 min. To a 10-ml aliquot in a polarographic vessel add 0·5N-KOH (0·06 ml), remove N by bubbling with O for 10 min. and register the polarographic wave ($E_{\frac{1}{2}}$ = −0·42 V, with a mercury-pool anode). Compare with a calibration curve of latex to which a known amount of tetraethylthiuram disulphide has been added. The wave due to S can be differentiated and causes no interference, neither does phenyl-1-naphthylamine, Na hexadodecylbenzenesulphonate, toluene or catechol, which are polarographically inactive in the medium used and may be present in minute amounts in the samples.
J. Zýka

3387. **Determination of anthraquinone in capacitor dielectrics.** P. D. Garn and M. C. Bott (Bell Telephone Lab. Inc., Murray Hill, N.J., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 84–85.—The anthraquinone is measured polarographically in $CHCl_3$–methanol (3:2) soln. containing either $MgCl_2$ or HCl. This solvent will dissolve a variety of dielectric materials. G. P. Cook

3388. **The use of ion-exchange methods in the analysis of waxes. II. Determination of neutral components.** W. Presting and S. Jänicke (Inst. f. Verfahrenstech. der organischen Chemie, Leipzig). *Fette, Seif., Anstrichmitt.*, 1961, **63** (1), 49–55 (in German).—Schemes are presented for the separation and analysis of the neutral components of waxes by means of cation exchange. Wax soaps and free wax acids are separated by mixed-bed ion exchange. It is possible to determine the total hydrocarbon waxes from the unsaponifiable matter, by a calculation which involves the amount extracted by butanone and the total eluate obtained by column chromatography of the unextracted material on SiO_2 at 85° in n-heptane. Results obtained from typical products are presented. S. M. Marsh

---

See also Abstracts—**3146**, Efficiency of oxidation reagents for methane. **3148**, Spot tests for organic compounds. **3274**, Titration of thiols. **3293**, Spot test for *p*-phenylenediamine. **3417**, Glucuronic and galacturonic acids. **3466**, Unsaturated aldehydes and ketones in foods. **3494**, Anionic surfactants in effluents. **3505**, Methane in gases.

4.—BIOCHEMISTRY

INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Biological fluids, animal and vegetable tissues

3389. **Complexometric estimation of potassium in erythrocytes.** W. Herbinger and W. Hubmayer (Gen. Hosp., Linz a.d. Donau, Austria). *Klin. Wochschr.*, 1960, **38** (20), 1064–1065.—Heparinised blood (2 to 3 ml) is centrifuged for 5 min. at 3000 r.p.m., and 0·1 ml of erythrocytes is transferred to 1·9 ml of water and haemolysed by mixing. The proteins are pptd. by addition of 1 ml of 5% trichloroacetic acid, and the ppt. is filtered off after 2 to 3 min.; 1 ml of the filtrate is then treated with sodium cobaltinitrite soln., and after standing for 5 min. the ppt. is separated by centrifuging. The ppt. is washed with 3 × 5 ml of water, by centrifuging, and dissolved in 3 drops of conc. H_2SO_4. This soln. is diluted with water, and 7 ml of 0·001M-EDTA (disodium salt) and 12 drops of aq. NH_3 (sp. gr. 0·910) are added, and the resulting soln. is titrated with 0·001M-$ZnSO_4$ to Eriochrome black T. The method was checked by a flame-photometric method, the difference between the methods being ±3·5%. D. B. PALMER

3390. **Determination of magnesium by the Titan yellow and ammonium phosphate methods.** F. W. Heaton (M.R.C. Unit for Metabolic Disturbances in Surgery, General Infirmary, Leeds). *J. Clin. Path.*, 1960, **13** (4), 358–360.—The Titan yellow (C.I. Direct Yellow 9) and ammonium phosphate pptn. methods for the micro-determination of Mg in a number of biological materials have been investigated, and some modifications to published methods are suggested. The Titan yellow method is quicker, but is subject to interference, and while reasonably satisfactory for serum and urine is unsuitable for the analysis of blood cells, faeces or food, and may give suspect results in pathological specimens of serum or urine. The ammonium phosphate pptn. method is more reliable and can be applied to a wide variety of materials with only slight modifications of the procedure, but takes much longer.
R. A. BRENAN

3391. **The determination of magnesium in biological fluids and tissues by flame spectrophotometry.** N. Alcock, I. MacIntyre and I. Radde (Postgraduate Medical School, Ducane Road, London, W.12). *J. Clin. Path.*, 1960, **13** (6), 506–510.—Methods for the determination of Mg in biological material with the Zeiss spectrophotometer PMQ II with flame attachment are described, and interference effects due to flame background, sodium emission and other constituents have been studied. Good recoveries were obtained, but it is necessary to use an oxy-acetylene flame, as oxy-hydrogen gives insufficient sensitivity. R. A. BRENAN

3392. **A spectrophotometric method of estimating blood oxygen using the Unicam SP.600.** D. Verel, R. Saynor and A. B. Kesteven (City General Hospital, Sheffield, England). *J. Clin. Path.*, 1960, **13** (4), 361–363.—The extinction of the blood specimen after haemolysing with saponin is read at 480 and 506 mμ in a special cell, the construction of which is described, and the blood oxygen is calculated with the aid of a nomogram. R. A. BRENAN

3393. **Diffusion - isothermic micro-determination of total nitrogen in urine.** I. S. Rolzman (Vinnitsky Med. Inst., USSR). *Lab. Delo*, 1961, **7** (1), 8–12.—*Procedure*—After the oxidation of a 1-ml sample of urine by the Kjeldahl method, dilute the colourless soln. to 50 ml and transfer a 5-ml aliquot (≡ 0·1 ml of urine) to one of the external chambers of a modified Conway apparatus. Introduce 30% NaOH soln. (3 ml) or satd. K_2CO_3 soln. into the other external chamber, and 0·05N-H_2SO_4 (2 ml) into the internal chamber. Cover, carefully mix the contents of the external chambers, and set the cup aside overnight. Use H_2O (1 ml) as a control. Add Tashiro indicator (1 to 2 drops) to the internal chamber and titrate the uncombined H_2SO_4 with 0·05N-NaOH to a colour change from lilac to pale green. Results obtained are satisfactorily reproducible by the usual Kjeldahl method. K. R. COOK

3394. **Determination of nitrogen in excreta.** I. K. Golovina and N. F. Koshelev (Kirov Military Med. Acad., USSR). *Lab. Delo*, 1961, **7** (1), 12–14.—*Procedure*—Add H_2O (100 to 200 ml) to faeces, and homogenise the mixture in a specially designed homogeniser, first at 4000 r.p.m. for 1 to 2 min. and then at 8000 r.p.m. for 1 to 2 min. To a weighed portion of the homogenate (1 g) add conc. H_2SO_4 (5 ml) and 30% H_2O_2 soln. (2 ml) and heat gently in a covered flask for 1 to 1·5 hr. until the contents are bleached. Cool, add H_2O (40 ml) and 0·05% aq. phenol red (2 drops), and neutralise. Add formaldehyde soln. (5 ml) and titrate to phenol red with 0·1N-NaOH. K. R. COOK

3395. **Determination of sulphur-35 in biological material.** K. Schmid (Forschungslab., CIBA Akt.-Ges., Basel, Switzerland). *Helv. Chim. Acta*, 1961, **44** (1), 84–88 (in German).—Weak β-emitters such as ^{35}S are subject to self-absorption; the sample must therefore be presented for measurement in a well-defined and reproducible form. Pptn. as $BaSO_4$ or benzidine sulphate is advantageous. For oxidation of the S, dry oxidation in a nickel bomb is suitable. *Procedure*—Weigh solid samples (500 mg or 1 millimole of S) direct into the bomb; weigh liquid samples into small pieces of aluminium foil and dry *in vacuo* before transfer to the bomb; cover moist samples with a thin layer of Na_2CO_3. Add ethanediol (6 drops) to the sample, cover with Na_2O_2 (5 to 10 g) and ignite. Then cool the bomb rapidly in water, and boil the contents with H_2O to extract and decompose the excess of Na_2O_2. Rinse out the bomb with H_2O (25 ml), add N-H_2SO_4 (2 ml) as carrier, and make the soln. weakly acid to methyl orange with HCl (20%), filter hot and wash the filter with hot H_2O (3 × 5 ml). Heat the filtrate to boiling and add $BaCl_2$ soln. (10%) (5 ml), dropwise. Allow to stand for 12 hr., collect the ppt. on a G4 sinter, and wash it first with hot H_2O (20 ml), then with ethanol and ether. Dry at 120° for 30 min., cool and weigh. For the counting, weigh 150 mg of the ppt. into a stainless-steel cup and press flat by a rotary action with a suitable tool. The ppt. prepared in this way is of so-called infinite thickness (40 mg per sq. cm), so that self-absorption corrections can be applied. Correct for the background count and the activity loss from the half-life and calculate the value for the sample. Recovery and reproducibility for samples ranging from [^{35}S]H_2SO_4 to [^{35}S]sulphaphenazole (the latter also with biological samples added) averaged 99·4 ± 0·7% for 76 measurements. Contamination of

the bomb by highly active substances was not observed. The preparation of ^{35}S-labelled compounds used is described. G. RUSSELL

3396. **Spectrophotometric determination of sulphate in serum and urine with barium chloranilate.** I. P. T. Häkkinen and L. M. Häkkinen (Univ., Turku, Finland). *Scand. J. Clin. Lab. Invest.*, 1959, **11** (3), 294–297.—To a 2-ml aliquot of the protein-free fluid is added 5 ml of benzidine soln. (1% in ethanol), and the mixture is allowed to stand for 1 to 2 hr. at 4° and centrifuged. The ppt. is dissolved in 2 ml of hot water and to the resulting soln. are added 95% ethanol (3 ml), acetate buffer (pH 4·0) (1 ml) and ≃30 mg of barium chloranilate. After shaking the mixture for 15 min., and separating the ppt. of barium sulphate and unchanged barium chloranilate by centrifuging, the extinction of the liberated chloranilic acid is measured at 530 mμ. Standards are prepared from Na_2SO_4 soln. This technique measures the serum inorganic sulphate. For the determination of the total serum sulphate, the ethereal sulphates are previously hydrolysed by heating with 0·1 ml of 2N-HCl at 80° for 2 hr. in a stoppered tube. For urine, 1 ml is treated with 1 ml of acetate buffer, 0·5 ml of potassium oxalate soln. (1 mg per ml), 3 ml of 95% ethanol, 6 ml of water and ≃30 mg of barium chloranilate. After shaking for 15 min. and filtering, the extinction of the liberated chloranilic acid is measured at 530 mμ. Blanks are essential to correct for urinary colour. Normal values for serum are reported as 2·4 to 3·3 mg per 100 ml (inorganic sulphate) and 2·7 to 3·9 mg per 100 ml (total sulphate). Both serum and urine values vary greatly with diet. The method is sensitive to 2 μg of sulphate per ml, and is suitable for routine use.
D. B. PALMER

3397. **Spectrophotometric determination of sulphate in tissue homogenates with barium chloranilate.** I. P. T. Häkkinen (Univ., Turku, Finland). *Scand. J. Clin. Lab. Invest.*, 1959, **11** (3), 298–300.—Tissue homogenate (0·5 ml) and 2N-HCl (0·5 ml) are kept overnight at 80° in order to hydrolyse the SO_4^{2-} conjugates. After adjustment of the pH to 4·0 with aq. NH_3, the method described for urine (*Anal. Abstr.*, 1961, **8**, 3396) is carried out.
D. B. PALMER

3398. **Use of the mercury-drop-electrode method for direct evaluation of minute amounts of lead in urine.** W. Kemula and Z. Kublik (Inst. of Phys. Chem., Polish Acad. Sci., Warsaw). *Nature*, 1961, **189**, 57–58.—The hanging-drop electrode previously described (*Anal. Chim. Acta*, 1958, **18**, 104; Kemula *et al.*, *J. Electroanal. Chem.*, 1959, **1**, 91) is used for the selective concentration of Pb before polarography. This method enables the Pb to be determined directly on normal urines as well as on those from cases of lead poisoning.
H. F. W. KIRKPATRICK

3399. **Determination of iron and copper in blood serum.** I. Dezső and T. Fülöp (Inst. für med. Chem. u. der I Med. Univ. Klinik, Debrecen, Hungary). *Mikrochim. Acta*, 1961, (1), 154–157 (in German).—Existing methods have been modified to provide a combined method for Fe and Cu. *Procedure*—Add 1·5 ml of 2N-HCl to 3 ml of serum; after 10 min. add 1·5 ml of 20% trichloroacetic acid soln. and centrifuge. For Fe, take a 2-ml aliquot of the centrifugate and add 0·5 ml of 2,2′-bipyridyl (**I**) soln. (dissolve 0·4 g of **I** in 5 ml of acetic acid, add 1 g of hydroxyammonium chloride and dilute to 100 ml) and 1·5 ml of 50% Na acetate soln. After 15 min. measure the extinction (Pulfrich photometer, filter S53). For Cu, take a 1-ml aliquot of the centrifugate and add 0·3 ml of 50% Na acetate soln. and 0·5 ml of 2·5% EDTA soln. Extract with 5 ml of Pb diethyldithiocarbamate soln. (*Anal. Abstr.*, 1960, **7**, 204) in n-butanol and measure the extinction (filter S43). The method is suitable for serial investigations.
P. D. PARR-RICHARD

3400. **Separation of cobalt, copper, manganese and iron and their determination in biological materials.** E. P. Kupaks. *Trudy Inst. Eksper. Med., Akad. Nauk SSSR*, 1960, **22**, 115–127; *Ref. Zhur., Khim., Biol. Khim.*, 1960, (21), Abstr. No. 29,472.—The following solvent systems are used for the separation by ascending paper chromatography of Co^{2+}, Cu^{2+}, Mn^{2+}, Ni^{2+} and Fe^{3+} in soln.: (*i*) isopropyl alcohol - acetone - HCl (sp. gr. 1·175) (15:2:8); (*ii*) isoamyl alcohol - acetone - HCl (sp. gr. 1·175) (4:5:1); (*iii*) n-propanol - acetone - HCl (sp. gr. 1·175) (65:2:33). It is shown that the R_F values for each type of ion depend on the temp., the solvent, the acid concn. of the soln. and the type of paper used. The separated ions can be determined by means of direct colorimetric measurement of the paper, or in soln. by the method of standard series. The best reagents to use are 2-nitroso-1-naphthol for Co^{2+}, and Na diethyldithiocarbamate (**I**) for Cu^{2+} and Mn^{2+}. After extraction from the paper and colorimetric measurement of the soln., good results are obtained with nitroso-R salt for Co^{2+}, **I** for Cu^{2+}, $(NH_4)_2S_2O_8$ for Mn^{2+}, and KSCN for Fe^{3+}. These methods are adapted to the determination of Co^{2+}, Cu^{2+}, Mn^{2+} and Fe^{3+} in biological materials where the ion concn. put on to paper is $\lesssim$0·5 μg.
K. R. COOK

3401. **Fluorimetric determination of uranium in urine.** L. Wódkiewicz (Dept. of Anal. Chem., Inst. of Nucl. Res., Warsaw, Poland). *Chem. Anal., Warsaw*, 1960, **5** (6), 985–992.—The sample is concentrated by distillation, mineralised with conc. HNO_3 and diluted with 5% HNO_3. This soln. is either (*a*) evaporated to dryness and fused with a mixture of NaF (9%), Na_2CO_3 (45·5%) and K_2CO_3 (45·5%), or (*b*) extracted with ethyl acetate, the ethyl acetate soln. being then evaporated and the residue fused as described above. The fluorescence of the sample is compared with that of a standard soln. of uranium nitrate. The error is $\gtrsim$5% for the direct determination and $\gtrsim$12% for the determination after ethyl acetate extraction; 10 μg of U per litre of urine can be determined.
B. K.

3402. **Separation of uranium from urine by a tri-n-octylphosphine oxide column and an automation of the procedure.** W. C. Dietrich, J. D. Caylor and E. E. Johnson (Union Carbide Nuclear Co., Y-12 Plant, Oak Ridge, Tenn., U.S.A.). *U.S. Atomic Energy Comm.*, Rep. Y-1322, 1960. 37 pp.—Uranium is adsorbed from partially digested urine on to a column of glass beads coated with tri-n-octylphosphine oxide, and removed from the column by eluting with ethanol. The stripped sample is evaporated, ashed and counted for α-activity. A machine performs the adsorption, stripping and evaporation sequences automatically. The average uranium recovery, which includes extraction efficiency, α-particle attenuation and counter variability, is 73%. NUCL. SCI. ABSTR.

3403. **Radiochemical determination of plutonium in urine.** H. V. Weiss and W. H. Shipman (Chem. Technol. Div., U.S. Navy Radiol. Def. Lab., San Francisco, Calif.). *Anal. Chem.*, 1961, **33** (1), 37–39.—A recovery of 93·7 ± 5% has been achieved in 5 to 6 hr. in control experiments with the method described, in which the plutonium is co-crystallised with potassium rhodizonate, then separated and freed from interfering substances by co-pptn. with lanthanum salts and by ion exchange. The plutonium is finally electro-deposited on a tantalum disc and α-counted. (*Cf.* Weiss and Lai, *Anal. Abstr.*, 1960, **7**, 4677.) E. G. CUMMINS

3404. **Radiochemical determination of radium in urine.** H. V. Weiss and M. G. Lai (Chem. Technol. Div., U.S. Naval Radiol. Def. Lab., San Francisco, Calif.). *Anal. Chem.*, 1961, **33** (1), 39–41.—Radium has been isolated from urine with a recovery of 94·9 ± 3·3% (11 determinations) by the method described, which is based on co-crystallisation with potassium rhodizonate and purification of the extract by ion exchange. E. G. CUMMINS

3405. **Analytical procedures at the U.S. Naval Radiological Defense Laboratory for the determination of certain radio-elements in urine.** W. H. Shipman and H. V. Weiss (Naval Radiol. Defense Lab., San Francisco, U.S.A.). *U.S. Atomic Energy Comm.*, Rep. USNRDL-TR-451, 1960. 26 pp.—Analytical procedures are described for the determination of the following radioactivities in urine—gross β-activity, ^{140}Ba, ^{90}Sr, Pu, Po, Ra and natural uranium. NUCL. SCI. ABSTR.

3406. **Determination of sulphonamides in biological material. Comparison of colorimetric and radiochemical methods on [^{35}S]sulphaphenazole.** W. Padowetz, K. Schmid and J. Druey (Forschungslab., CIBA Akt.-Ges., Basel, Switzerland). *Helv. Chim. Acta*, 1961, **44** (1), 89–94 (in German).—The colorimetric method is essentially that of Bratton and Marshall (*J. Biol. Chem.*, 1939, **128**, 537), and radiochemical determinations were carried out as described for [^{35}S]sulphaphenazole (Schmid, *Anal. Abstr.*, 1961, **8**, 3395). The mean error was ±1%. A simplified technique can sometimes be used with blood, plasma and tissues, by which samples are lyophilised on to aluminium plates and counted direct. In another procedure for blood, shake the sample (1 to 2 ml) with ethyl acetate (10 ml) for 20 min. and chromatograph aliquots on Whatman No. 1 paper with Bush C solvent. Stick the zones on to aluminium plates and measure the activity; the R_F for sulphaphenazole is 0·54, and for the acetyl derivative 0·31. With urine samples, the colorimetric and radiochemical methods gave similar results. With blood, if the values of the colorimetric method were corrected from a calibration curve prepared by known additions of sulphaphenazole to blood (especially necessary if acetylsulphaphenazole is present), reasonable agreement was obtained. Correction for sulphaphenazole gave only 88·6 ± 8·5% of the radiochemical value; correction for acetylsulphaphenazole as well gave 102·4 ± 19·2% of this value. Serum and plasma were less prone to protein error than blood. Tissues caused difficulties at the extraction stage and comparison could not be made. G. RUSSELL

3407. **Chemotherapy of intestinal infections. III. Estimation of *N*4-acyl derivatives of sulphonamides in animal tissues.** V. M. Patki and M. V. Shirsat (Dept. of Chemotherapy, Haffkine Inst., Bombay). *J. Sci. Ind. Res., India, C*, 1961, **20** (1), 18–20.—The procedure of Bratton and Marshall (*J. Biol. Chem.*, 1939, **128**, 537) for the determination of sulphonamides has been modified to permit the determination of the N^4-acyl derivatives. The tissue is extracted with 0·01N-NaOH in 50% ethanol in a Waring blender, the extract and washings being made up to 100 ml. *Determination of free sulphonamide*—A mixture of extract (5 ml), 15% trichloroacetic acid soln. (4 ml), water (1 ml) and ethanol (10 ml) is set aside for 15 min. and then filtered. To the filtrate (10 ml) are added 4N-HCl (0·5 ml) and 0·1% $NaNO_2$ soln. (1·0 ml) followed, after 3 min., by 0·5% ammonium sulphamate soln. (1·0 ml) and, after a further 2 min., 0·1% *N*-1-naphthylethylenediamine dihydrochloride soln. (1 ml) and water (1·5 ml) are added. The extinction is read at 550 mμ, and results are referred to a standard curve prepared from sulphadiazine. *Determination of total sulphonamido-compounds*—The extract is diluted with 0·01N-NaOH in 50% ethanol so as to contain 8 to 10 μg per ml of free sulphonamide. The diluted extract (10 ml) is heated under reflux on a boiling-water bath with 4N-HCl (1·5 ml) for 2 hr. After cooling, the mixture is made up to volume and filtered if necessary; 4N-HCl (0·5 ml), butanol (1·5 ml), 0·1% $NaNO_2$ soln. (1 ml), 0·5% ammonium sulphamate soln. (1 ml) and Bratton and Marshall reagent (1 ml) are added as described above. The addition of butanol is necessary to keep released lipids in soln., but it is not necessary to add trichloroacetic acid to precipitate proteins. Recoveries of ≃90% are obtained by the modified procedure. R. A. BRENAN

3408. **A modified extraction method for determination of Evans blue [azovan blue].** S. Jarnum (Bispebjerg Hospital, Copenhagen, Denmark). *Scand. J. Clin. Lab. Invest.*, 1959, **11** (4), 332–339.—A modification of Allen's procedure (*Amer. J. Physiol.*, 1953, **175**, 227) is described, the principle of which is the splitting of the dye - protein complex with a detergent, and subsequent determination of the released dye by photometry. Heparinised plasma or serum (4 ml) is mixed with aq. sodium lauryl sulphate (10%) (5 ml) and paper-pulp (0·25 g). The mixture is shaken for 0·5 to 1 min., then transferred to a tube and the paper column is allowed to drain. The column is washed twice with normal saline (3 to 5 ml) and then with methanol - normal saline (9:1). After acidification with 0·1N-HCl, the adsorbed dye is eluted with 0·2N-HCl - acetone (1:1) (5 ml) and the column is washed with a further 5 ml of acid acetone. The combined eluates are made up to 10 ml and the extinction of the soln. is measured at 620 mμ. As a blank, water replaces plasma or serum, and a 1% aq. soln. of azovan blue is used as a standard. An average recovery of 92·5% is obtained. D. B. PALMER

3409. **A specific sensitive and rapid micro-method for determination of ketone bodies in blood.** O. Hansen (Mecodumex, Dumex Ltd., Copenhagen, Denmark). *Scand. J. Clin. Lab. Invest.*, 1959, **11** (3), 259–268.—The method of Greenberg and Lester (*J. Biol. Chem.*, 1944, **154**, 177, and 1948, **174**, 903) was fully investigated, and it was found that reproducible results could not be obtained by its use. However, the basic principle of the method, oxidation to acetone and reaction with 2,4-dinitrophenylhydrazine and measurement of the extinction of the corresponding hydrazone (in alkaline soln.) in CCl_4, provided the basis for an extremely sensitive and

accurate method, which is described in detail. Glucose (2%) and lactic acid had no effect on the method. Satisfactory recoveries were obtained when pure aq. soln. of acetone and of β-hydroxybutyric acid were used (99·2 to 100% and 99·6 to 106·3%, respectively). The recovery of acetone from blood was 99·4 to 100%. To determine β-hydroxybutyric acid separately, the ketone bodies are determined with and without the addition of $K_2Cr_2O_7$ in the distillation process. The difference between these two values gives the amount of β-hydroxybutyric acid as acetone. D. B. PALMER

3410. **Detection of reducing sugars in urine specimens by paper chromatography.** S. N. Tewari [18/3A The Mall, Kanpor (O.P.), India]. *Mikrochim. Acta*, 1961, (1), 37–40 (in English).—*Cf. Z. anal. Chem.*, 1960, **176** (6), 406; *Anal. Abstr.*, 1961, **8**, 2073.

P. D. PARR-RICHARD

3411. **Rapid method for true blood glucose estimation.** O. M. Forsell and I. P. Palva (Centr. Hosp. of Northern Carelia, Joensuu, Finland). *Scand. J. Clin. Lab. Invest.*, 1959, **11** (4), 409.—The method of Athanail and Gabaud (*cf. Anal. Abstr.*, 1958, **5**, 3830) is adapted for routine use by carrying out the reaction at increased pressure. Serum protein is pptd. by NaOH - $ZnSO_4$, 2-biphenylamine is added to the protein-free filtrate, and the reaction mixture is heated in stoppered tubes for 10 min. at 2 atm. in a pressure cooker. The extinction of the condensation product is then measured at 650 mμ. This procedure is simple and rapid, and requires the use of only one stable reagent.

D. B. PALMER

3412. **Enzymatic determination of glucose. Stabilisation of colour developed by oxidation of o-dianisidine.** G. Guidotti, J.-P. Colombo and P. P. Foà (Chicago Med. Sch., Ill., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 151–153.—The colour stability was improved by modification of the pH of the soln. (to $<3·0$ or to $>10·0$), by addition of gum ghatti, or by extraction of the colour into isobutyl alcohol. By these means a standard deviation of $<\pm 2\%$ was achieved. G. P. COOK

3413. **Determination of dehydroascorbic and ascorbic acids in urine.** A. P. Rŷzhova ('Barvikha' Sanatorium, Min. of Public Health, USSR). *Lab. Delo*, 1961, **7** (1), 14–18.—*Procedure*—For the calibration curve, oxidise ascorbic acid (**I**) to dehydroascorbic acid (**II**) by adding bentonite (1·5 to 2·0 g) to **I** soln. (100 mg of **I** per 100 ml of stabilising soln. *A*) (20 ml), shake for 10 min. and filter. Prepare a series of tubes containing 5 to 40 μg of **I** and add freshly prepared soln. *A* [2N-HCl (1 ml) and 0·1% thiourea soln. (3 ml), diluted to 400 ml with H_2O] to a vol. in each tube of 2 ml. Add 5% thiourea soln. (1 drop) and 2% 2,4-dinitrophenylhydrazine soln. (0·5 ml) to each tube. Shake the tubes, stopper them and place them in a boiling-water bath for 10 min. Cool rapidly in water and ice, then make up to 5 ml with 85% H_2SO_4. Stir well after each addition. Measure the reddish colour photometrically against a control after >1 hr. and plot a calibration curve. The curve is rectilinear and the system obeys the Beer - Lambert law. To a sample of fresh urine (15 ml) add a stabilising mixture of 0·1% aq. thiourea soln. (3 ml) and 2N-HCl (1 ml); store at 1° to 5°. Add H_2O (38 ml) to the filtered, preserved urine (2 ml). To determine **II** in urine use unoxidised urine. To determine **I** plus **II**, add bentonite (1·5 to 2 g) to the urine soln. (20 ml), shake for 10 min. and filter. Take a 2-ml aliquot of urine soln., or urine filtrate obtained after oxidation with bentonite, and continue as for the calibration curve. Use soln. *A* (2 ml) for the control. K. R. COOK

3414. **Determination of oxalic acid in urine.** A. Hodgkinson and P. M. Zarembski (Med. Res. Council Unit for Metabolic Disturbances in Surgery, General Infirmary, Great George Street, Leeds, England). *Analyst*, 1961, **86**, 16–21.—The urine (50 ml) is mixed with 19 g of $(NH_4)_2SO_4$ and 5 ml of conc. HCl, the change in vol. being noted. After 30 min. an aliquot (20 ml) of the filtered liquid is extracted for 6 hr. with ethyl ether in a special apparatus (described). Water (4 ml) is added to the extract and the ether is removed by evaporation. The aq. residue is centrifuged with three 2-ml portions of ethanol and the apparatus is washed with 2 ml of water. Aq. NH_3 (50% v/v) is added until the liquid is alkaline to bromocresol green. Calcium oxalate is pptd. with $CaCl_2$ soln. under specified conditions in the presence of acetic acid and, after the ppt. has settled overnight, the supernatant liquid is removed, the washed ppt. is dissolved in 2N-H_2SO_4 and the soln. is heated in a water bath with zinc powder. An aliquot of the soln. of glycollic acid so formed is heated with chromotropic acid and H_2SO_4 in a water bath, and the extinction of the cooled liquid is measured at 570 mμ and referred to a calibration graph prepared by reducing standard oxalic acid soln. in the same manner. The recovery was 97 to 100% (mean 98·1 ± 2·0%). A. O. JONES

3415. **Method for the isolation and estimation of furan-2,5-dicarboxylic acid in human urine.** Samiha M. Abdel Wahhab (Chem. Dept., Fac. Sci., Alexandria Univ., Egypt). *Mikrochim. Acta*, 1961, (1), 75–81 (in English).—When Ca gluconate and inositol were added to samples of urine no increase in the amount of furan-2,5-dicarboxylic acid (**I**) was detected, confirming that **I** is formed *in vivo* from these substances, but not *in vitro* under the conditions of test. A modified method for isolating and determining **I**, in which various acid treatments are omitted, further supports this view, and recoveries of **I** were higher (mean value 91·16%) than those obtained by the original test procedure (Flaschenträger and Wahhab, *Ibid.*, 1950, 275).

P. D. PARR-RICHARD

3416. **Determination of glucuronic acid.** J. Marogg (Med. Klin., Kantonspitals St. Gallen, Switzerland). *Arzneimittel-Forsch.*, 1960, **10** (12), 987–990.—Methods of determination are critically reviewed, and a method for application to biological fluids is presented. *Procedure*—Mix the sample (2 ml) in 0·9% NaCl soln. (14 ml) with 10% Na_2WO_4 soln. (2 ml) and 0·66N-H_2SO_4 (2 ml), and filter after 10 min. To the filtrate (2·5 ml) add 2 ml of reagent [Na_2CO_3 - $NaHCO_3$ buffer (pH 10·1) (2·05 ml), aq. iodine - KI soln. (5% of iodine and 10% of KI) (1·5 ml), 10·4% $NaHSO_4$ soln. (0·15 ml) and 17% H_2SO_4 (0·3 ml)], followed by 0·4% 1,3-dihydroxynaphthalene soln. in 95%

ethanol (2 ml) and 50% H_2SO_4 (2 ml). Heat at 100° for 1 hr., cool for 10 min. and add 95% ethanol (5 ml). Shake the mixture for 3 min., add toluene (8 ml), mix well and set aside in the dark for 2 hr., then measure the extinction of the toluene layer at 565 mμ. To determine glucuronic acid without removal of protein, mix the sample (1 ml) with 0·9% NaCl soln. (29 ml) and treat 2·5 ml of the mixture as described above for the filtrate from the Na_2WO_4 pptn. A. G. COOPER

3417. **Micro-analysis of glucuronic and galacturonic acids.** Hisaaki Toda. *J. Soc. Text. and Cellulose Ind., Japan*, 1960, **16** (9), 719–724 (in Japanese, English summary).—Qualitative and quant. determinations of uronic acids (**I**) and uronides were performed by conductimetric and potentiometric titrations and by paper chromatography. The dissociation of glucuronic acid lactone is very slow in neutral and acid soln. On titration with $Ba(OH)_2$ soln., the pH increases rapidly and the soln. becomes alkaline before the lactone begins to dissociate. By contrast, **I** in the acid form react immediately with the hydroxide and form barium uronates below pH 7. Hence the proportion of **I** to their lactones can be determined by a combination of conductimetric and potentiometric titrations. The paper chromatography of **I** and their lactones in the presence of other sugars was carried out by multiple development of their barium salts with n-butanol - benzene - pyridine - water (10:2:5:5) and ethyl acetate - acetic acid - formic acid - water (18:3:1:4), followed by colorimetric determination of the **I** fractions that separated clearly from the other neutral sugars. Although the uronides of gum acacia and pectin are highly resistant to hydrolysis, a significant proportion was hydrolysed to monomers by prolonging the second hydrolysis to 10 to 12 hr. The decomposition of **I** during hydrolysis was studied, and the **I** content of gum acacia and pectin was determined by chromatography. The uronide contents obtained agreed closely with the results of the carbon dioxide method. ABSTR. BULL. INST. PAPER CHEM.

3418. **Hydroxamic acids. X. Micro-determination of acetyl groups in pectins.** E. E. Vonesch, O. A. Guagnini and M. H. C. K. de Riveros (Univ. Buenos Aires, Argentina). *An. Asoc. Quim. Argentina*, 1960, **48** (2), 117–123.—The pectin (20 to 40 μg) is treated with aq. NaOH to hydrolyse the acetyl groups, the acetate is converted into ethyl acetate with ethanol - H_2SO_4 and the ester is continuously distilled in a current of air at >80°. The distilled ester is collected in alkaline hydroxylamine soln. and the acetohydroxamic acid, after treatment with $Fe(ClO_4)_3$, is determined photometrically as the ferric chelate. Comparisons with a direct hydroxylaminolysis method have been made. L. A. O'NEILL

3419. **Rapid, simple colorimetric method for the determination of micro quantities of gibberellic acid.** H. D. Graham and L. B. Thomas (The George Washington Carver Foundation, Tuskegee Inst., Alabama, U.S.A.). *J. Pharm. Sci.*, 1961, **50** (1), 44–48.—Evaporate an aq. soln. of gibberellic acid or gibberellin (40 to 400 μg) by heating at 105° for 2 hr. Dissolve the residue in carbonyl-free ethanol (1 ml), add 1 ml of reagent [heat 2,4-dinitrophenylhydrazine (100 mg) with carbonyl-free ethanol (50 ml) and conc. HCl (5 ml) until dissolved, cool and dilute to 100 ml with carbonyl-free ethanol], and heat at 100° for exactly 5 min. Cool in ice for 5 min., add a 10% soln. of KOH in 80% carbonyl-free methanol (5 ml), mix well and set aside for 5 min. Add H_2O (15 ml), mix well, dilute 1:2 with H_2O and measure the extinction at 430 or 540 mμ against a reagent blank. Indol-3-ylacetic acid, 2,4-dichlorophenoxyacetic acid and other plant auxins do not give the characteristic wine-red colour. Tween 20 and other polyoxyethylene-type compounds interfere. A. R. ROGERS

3420. **Colorimetric determination of gibberellic acid.** G. S. Muromtsev and M. N. Hestyuk. *Vestn. Sel.-khoz. Nauk.*, 1960, (2), 119–122; *Ref. Zhur., Khim., Biol. Khim.*, 1960, (19), Abstr. No. 26,643.—*Procedure*—Add Folin - Ciocalteu reagent (0·5 ml) to the test soln. (1 ml), stir carefully, add conc. HCl (2·5 ml) and stir again. Measure the green colour, which appears slowly, after 15 min. in a 1-cm cell with a red filter. For the control (greenish yellow) use a mixture of H_2O (2 ml), reagent (1 ml) and conc. HCl (5 ml); 10 to 200 μg of gibberellic acid per ml can be determined. K. R. COOK

3421. **Determination of flavonoid compounds after their separation by paper chromatography.** J. Davídek (Res. Inst. of Food Ind., Prague). *Biokhimiya*, 1961, **26** (1), 93–98.—*Procedure*—To Whatman No. 1 paper apply soln. (5, 10, 15, 20 μl) having concn. of 4 μg of rutin or quercitrin per μl of methanol and the same concn. of naringin in ethanol and develop for 10 hr. by the ascending technique with n-butanol - acetic acid - H_2O (20:5:11). Locate the zones of the dry chromatogram under u.v. light or by treatment with ammonia fumes. Section the chromatograms and elute each section in a test-tube by shaking with 5 ml of acid - methanol (1:1) for rutin or quercitrin or acid - ethanol (1:1) for naringin; the acid should be 0·2N-HCl or 0·2N-H_2SO_4. Elution is complete after 8 to 20 hr. according to the flavonoid compound present. For the colorimetric determination, add 0·5% *p*-aminobenzoic acid (0·4 ml), 10% H_2SO_4 (0·4 ml), 0·2% $NaNO_2$ soln. (2 ml) and 10% NaOH soln. (5 ml) and shake again. Measure the extinction of the soln. at 420 mμ (immediately for rutin and quercitrin and after 20 min. for naringin). This method is convenient for samples with a low flavonoid content. K. R. COOK

3422. **Quantitative estimation of ipomeamarone by chromatostrip technique.** Takashi Akazawa and Kajiro Wada (Lab. of Biochem., Fac. of Agric., Nagoya Univ., Anjô, Aichi). *Agric. Biol. Chem., Japan*, 1961, **25** (1), 30–33.—Ipomeamarone (**I**), which is of importance as a product of infection of sweet potatoes by black rot fungus, may be determined in micro amounts by separation on silica-gel chromatostrips, followed by elution and colorimetry with 2,4-dinitrophenylhydrazine by the method of Lappin and Clark (*Anal. Chem.*, 1951, **23**, 541). The sample is prepared by macerating 1 g of undried material with methanol (5 ml) and making up to 10 ml. An aliquot (0·2 ml) is applied to the chromatostrip and developed with ethyl acetate (10%) in n-hexane. Recoveries of **I** are satisfactory (90%) provided that the extract is analysed immediately after preparation. J. R. HUDSON

3423. **Determination of microgram amounts of sulphur-containing compounds [in plants].** E. J. Skerrett and G. J. Dickes (Res. Sta., Long Ashton, Bristol, England). *Analyst*, 1961, **86**, 69–71.—

Since anomalous results occurred in the determination of microgram amounts of lime-sulphur deposits on plants by the method in which evolved H_2S is converted into methylene blue by reaction with *NN*-dimethylphenylenediamine (**I**), the reaction conditions were examined and a satisfactory method was developed. The sample (containing $\geqslant 15$ μg of S and 1 g of water) is placed in an apparatus consisting of a flask fitted with an inlet tube for N and a vertical condenser connected by means of a detachable tube with a graduated 25-ml receiver. A reducing mixture of formic acid, HI and red phosphorus (freed from H_2S by prolonged passage of N at b.p.) is added and a soln. containing Zn acetate, Na acetate and acetic acid is placed in the receiver. Nitrogen is passed through the apparatus and the liquid is heated to b.p. during 5 min. and gently boiled for a further 15 min. The detached delivery tube is dropped into the receiver and 3 ml of a chromatographically purified soln. of **I** in dil. HCl is added. After 10 min., 0·2 ml of a $FeCl_3$ soln. is added and after 15 min. the rinsed delivery tube is removed, the liquid is made up to 25 ml and its extinction is measured at 670 mμ. The calibration graph is prepared from K_2SO_4 soln. A. O. JONES

3424. **Analysis of iron chelates in plant extracts.** D. G. Hill-Cottingham and C. P. Lloyd-Jones (Res. Sta., Long Ashton, Bristol). *J. Sci. Food Agric.*, 1961, **12** (1), 69–74.—Methods are described for the identification and quant. determination of EDTA (**I**) and 1,2-diaminocyclohexanetetra-acetic acid (**II**) or their Fe chelates. These depend upon the removal of unwanted plant constituents and concentration of the chelates by passage through a column of cation-exchange resin or of activated charcoal, followed by evaporation and paper chromatography with phenol - water (4:1 w/v) in the presence of aq. NH_3 and KCN. R_F values are given. For the quant. determination of **I** and **II**, colorimetric methods depending on either the Fe^{III} - salicylate complex or the Cu - biscyclohexanone oxalyldihydrazone complex are suggested. The Fe chelates of **I** and **II** may be determined by a modified 1,10-phenanthroline method; up to 200 μg can be determined. M. A. ELLIS

3425. **Quantitative paper electrophoresis of mixtures of porphyrins.** H. Ippen and H. Schlink (Hautklin. Med. Akad., Düsseldorf). *Arzneimittel-Forsch.*, 1960, **10** (12), 1034–1035.—The sample soln. is acidified with acetic acid and boiled with fresh ferrous acetate soln.; the resulting complexes are pptd. with talc and then extracted into aq. NH_3. After paper-electrophoretic separation in barbitone buffer soln., porphyrins are detected by treating the paper with 2·5% benzidine soln. in 25% methanolic acetic acid, 0·3% methanolic H_2O_2 soln. and 3% $Al_2(SO_4)_3.(NH_4)_2SO_4.24H_2O$ soln., drying in air and rendering translucent with 1-bromonaphthalene - liquid paraffin. The porphyrins appear as deep-blue to grey spots. A. G. COOPER

3426. **Silicon tetrachloride-treated paper in the paper chromatography of phosphatides. IVA, IVB. Origin of the gradient and factors influencing the course of the gradient curve.** H. G. Bungenberg de Jong and J. T. Hoogeveen (Dept. of Med. Chem., Univ. of Leyden, Netherlands). *Proc. Kon. Ned. Akad., Wet. B*, 1961, **64** (1), 1–17; 18–33 (in English).—Phosphatides studied were lecithin, cephalin, sphingomyelin, lysolecithin and lysocephalin as 0·5% or 1% soln. in $CHCl_3$ - methanol (4:1); $\simeq 5$ μl of soln. was applied. The solvent for chromatography was di-isobutyl ketone - acetic acid - H_2O (10:5:1 or 8:5:1). Chromatography was carried out in a dark room at 20°, with S. & S. 2043b paper. Paper treated with $SiCl_4$ was either exposed to air for different times (to remove HCl) or was neutralised or washed. The spots were detected with acid fuchsine - uranyl nitrate - HCl. With these conditions fixed, some factors were systematically investigated. Better separation was achieved with a larger air space in the tank, and this was correlated with a decrease in the gradient of acetic acid concn. along the paper. This applied with both untreated and treated paper. Descending chromatography gave better separation than ascending, although the results were less reproducible with the former technique. Since ascending chromatography is usually slower, this was expected to lead to a decrease in acetic acid gradient (by diffusion of vapour) which should give better separation. The explanation given assumes the diffusion factor to be outweighed by convection or rotational vapour effects. The effect of paper-fibre direction was studied. Better separation was achieved by developing the chromatogram in the direction of the slower solvent movement. This was ascribed to a longer time being available for decreasing the slope of the acetic acid gradient, which leads to better resolution. The effect of stirring, by means of glass strips actuated through the front and back of the tank, was examined. One stirrer was almost as effective as two in opposite directions (50 r.p.m.). Better separation was obtained by using stirring, and again this was ascribed to the effect on solvent gradient. The positions of the spots in the two cases corresponded fairly well with positions of the same acetic acid concn., although the R_F values were different. Increase in temp. generally improved separation, with a much larger change from 4° to 20° than from 20° to 25°. In some cases at 25°, separation was a little poorer. Many factors are listed as entering into the temp. effect, and the over-all effect cannot be predicted. G. RUSSELL

3427. **Colorimetric determination of isoleucine in protein hydrolysates.** B. Bulhak and M. Szelagiewicz (Dept. of Biochem., Univ., Warsaw). *Chem. Anal., Warsaw*, 1961, **6** (1), 83–89.—A specific method, permitting the determination of isoleucine in soln. containing leucine and valine, has been developed by combining the methods of Block *et al.* (*Proc. Soc. Exp. Biol. Med.*, 1940, **45**, 289; "The Amino-Acid Composition of Proteins and Foods," Springfield, 1951) and Albanese and Irby (*Brit. Abstr. C*, 1948, 272). The major modifications are—(*i*) the de-amination of isoleucine is carried out at room temp. for 12 hr.; (*ii*) the oxidation is performed with $KMnO_4$ in a phosphate buffer at pH 6·8 with simultaneous distillation of ethyl methyl ketone in a described apparatus; and (*iii*) the compound formed by ethyl methyl ketone and vanillin is diluted with ethyl acetate before being determined colorimetrically. The average yield of the oxidation product is 54 ± 0·5%, and a straight-line relationship between extinction and concn. obtains for 5 to 20 mg of isoleucine (≡0·06 to 0·24 mg of ethyl methyl ketone). The extinction is measured at $\simeq 600$ mμ. P. BRYCH

3428. **Separation and determination of tyrosine and 3,4-dihydroxyphenyl-L-alanine [DOPA] on ion-exchange resins. Application to the determination of DOPA in proteins oxidised by polyphenoloxidase.**

M. Rolland, S. Lasry and S. Lissitzky (Biol. Chem. Lab., Fac. Med., Marseilles). *Bull. Soc. Chim. Biol.*, 1960, **42** (9–10), 1065–1077.—Dowex 50-X4 (200 to 400 mesh) is treated by the method of Moore and Stein (*J. Biol. Chem.*, 1951, **192**, 663), by which it is converted first into the Na^+ form and then into the NH_4^+ form, in which state it is stored under water. Before use the resin is equilibrated by passage of 300 ml of ammonium acetate buffer (0·2M, pH 5·2) over a period of 12 to 24 hr. The mixture to be separated (1 to 6 ml, pH 4·0 to 4·5) is deposited and allowed to adsorb on the resin, which is then washed with 2 ml of ammonium acetate buffer (0·2M, pH 4·0 to 4·48); the elution is then carried out with 300 ml of ammonium acetate buffer (0·4M, pH 5·64) at a rate of 2 ml per 5 to 8 min., collecting fractions of 2 ml. The resin can be regenerated by treating it as described above. Analysis of the eluates is by either of 2 methods: (*a*) spectrophotometry at 279 mμ for 3-(3,4-dihydroxyphenyl)-L-alanine (**I**), and at 275 mμ for tyrosine; (*b*) colorimetry with the 1-nitrosonaphthol method of Udenfriend and Cooper (*J. Biol. Chem.*, 1952, **196**, 227) for tyrosine, and the nitrite - molybdate method of Arnow (*J. Biol. Chem.*, 1937, **118**, 454), modified to give increased sensitivity, for **I**. The results obtained by both methods are in good agreement, and it is possible to determine 50 to 200 μg of **I**, and 50 to 400 μg of tyrosine. D. B. PALMER

3429. **Estimation of cystine plus cysteine in proteins by the disulphide interchange reaction.** A. N. Glazer and E. L. Smith (Coll. of Med., Univ. of Utah, Salt Lake City, U.S.A.). *J. Biol. Chem.*, 1961, **236** (2), 416–421.—The interchange reaction in strongly acid soln. between *NN'*-di-2,4-dinitrophenyl-L-cystine (**I**) and cystine, simple thiols and various proteins is studied, and the effects of concn. of acid, temp., and concn. of protein are determined. The equilibrium values of the reaction are used in a method for the determination of the half-cystine plus cysteine content of proteins, in which the protein, dissolved in 9·6N-HCl, is incubated with excess of **I** at 39°; aliquots are removed at intervals, and after the unchanged **I** has been extracted with ethyl ether the mixed disulphides present in the aq. phase are determined spectrophotometrically at 357 mμ. J. N. ASHLEY

3430. **Some findings on the staining of serum proteins separated by paper electrophoresis.** F. Pipino (Orthopaedic Clin. Inst., Univ. of Bari, Italy). *Ital. J. Biochem.* (English Edition), 1960, **9** (5), 290–295.—Dyes commonly used to stain protein bands [Amido black 10B (C.I. Acid Black 1), azocarmine B] are taken up to different extents by albumin and globulins and the background usually retains some of the dye. Molybdenum blue is preferred since a more uniform staining of the proteins and the complete decolorisation of the electropherogram between the fractions are readily attained. *Reagent*—Add quinol (10 g) and Na_2SO_3 (anhyd.) (5 g) to a saturated soln. of molybdophosphoric acid in methanol - water (1:1) (100 ml). *Procedure*—Separate the serum (20 μl) on Whatman No. 1 paper at pH 8·6, at 120 V, for 12 hr. Dry the strip at 80° and immerse it first in trichloroacetic acid soln. (20%) for 5 min. and then in the reagent for 5 to 10 min., then wash it in tap water. Dry and render the zones transparent with liquid paraffin bromonaphthalene (1:1) and measure by the usual method. Comparative results are given for the 3 dyes with 30 normal sera. G. RUSSELL

3431. **Estimation of plasma fibrinogen, using sodium sulphite fractionation.** J. F. Goodwin Wayne County Gen. Hosp., Eloise, Mich.). *Amer. J. Clin. Path.*, 1961, **35** (3), 227–232.—Oxalated or heparinised plasma (1·0 ml) and 13% Na_2SO_3 soln. (9·0 ml) are mixed in a tube marked at 8 or 10 ml and placed in a water bath at 37° for 10 min. After centrifuging for 5 to 10 min. at 3000 r.p.m. the supernatant fluid is decanted off and the tube drained for 1 min. Sodium sulphite soln. (5 ml) is blown into the tube to break up the ppt. and after shaking vigorously a further 3 ml is added, washing down the sides of the tube, which is then re-centrifuged and drained as before. The ppt. is dissolved in 5% $Na_2PO_4.12H_2O$ (8 or 10 ml) with the aid of heat and after cooling the soln. it is made up to 8 or 10 ml again. A soln. of $CuSO_4.5H_2O$ (4%) (0·2 ml) is added, the tube is shaken vigorously for 2 or 3 min. and centrifuged for 10 min. at 3000 r.p.m. The extinction of the supernatant fluid is read at 560 mμ against a water blank. Standard protein soln. (1·0 ml) are mixed with Na_2PO_4 soln. (7 or 9 ml) and $CuSO_4$ soln. (0·2 ml) and treated as above. The method was compared with a more tedious thrombin pptn. procedure, and good agreement was obtained. R. A. BRENAN

3432. **Determination of the total acid-soluble fraction of animal tissues.** [**Determination of nucleotides.**] C. M. Calderera and M. Marchetti (Ist. Chim. Biol., Univ. Bologna, Italy). *Biochim. Appl.*, 1960, **7** (6), 332–336.—Freshly removed chicken livers are immediately frozen at −20° and a 20% homogenate in H_2O is prepared at 0° from an aliquot. A $HClO_4$ extract of the homogenate is neutralised with N-KOH and the resulting ppt. removed. The supernatant liquid is mixed with 0·05M-tri(hydroxymethyl)methylamine hydrochloride (pH 7·8). The buffered soln. is mixed with Dowex 2-X8 resin (200 to 400 mesh), activated by seven successive alternate treatments with N-HCl and N-NaOH followed by 4 treatments with N-HCl then washing free from Cl. The amount of resin should be 1 mg per 1 μg of nucleotides. After contact for 1 hr. the resin is filtered off, washed with the buffer soln. and extracted 5 times for 30 min. with 1·5N-HCl at room temp. The extinction at 260 mμ is measured on an aliquot of the combined extracts. A soln. of purified ribonucleic acid, hydrolysed by the method of Schmidt and Tannhauser (*J. Biol. Chem.*, 1945, **161**, 83), is used as a standard. J. I. M. JONES

3433. **Colorimetric determination of deoxyribonucleic acid in animal tissue and micro-organisms.** A. V. Andryushechkina, L. P. Nikolaeva and S. L. Freidman (Med. Inst., Saratov, USSR). *Lab. Delo*, 1961, **7** (1), 7–8.—*Procedure*—Centrifuge (× 2) the sample (250 mg) with H_2O for 10 min. each time at 1500 to 2000 r.p.m. Hydrolyse the washed material with N-HCl (5 ml) and stand the centrifuge tube in a water bath at 60° for 10 min. Add 1% methyl green soln. (10 ml) dropwise to the centrifugate while stirring, set the tube aside at 37° for 1·5 hr. and measure the intense yellow - brown colour obtained, with filter No. 4. Treat standard deoxyribonucleic acid (**I**) (1·5 to 2 mg) similarly. Methyl green is bleached by **I** so that the lower the colour intensity the higher the **I** concn. in a given sample. K. R. COOK

3434. **Detection of biogenic amines on paper chromatograms.** M. A. Iorio, F. Giora and G. B. Marini-Bettòlo (Lab. di Chim. Terapeutica, Ist.

Sup. di Sanità, Roma, Italy). *J. Chromatography*, 1961, **5** (1), 82–84 (in English).—Dip the chromatogram in unbuffered aq. 0·5% $K_3Fe(CN)_6$ soln. for 10 min., dry in an oven and dip rapidly in 0·5% $FeCl_3$ soln. Blue spots form on a yellow background. The sensitivity of the test is in the range 0·1 to 0·5 μg. The colour is given by many amines which are not revealed by $FeCl_3$ if buffered $K_3Fe(CN)_6$ soln. is used (*cf.* Goldenberg *et al.*, *Science*, 1949, **109**, 534). A positive reaction is also given by catechol, resorcinol, quinol, ascorbic acid and other reducing compounds. The amines tested include some synthetic compounds related to adrenaline as well as naturally occurring compounds. A. R. ROGERS

3435. **Quantitative chromatography and electrophoresis of ribonucleic acid nucleotides in tissue. Chemical relations between ribo- and deoxyribonucleic acids.** J. Montreuil, P. Derumez and P. Boulanger (Fac. de Méd. et de Pharm., Lille, France). *Compt. Rend.*, 1960, **251** (25), 3100–3102.—The procedure given avoids an initial separation of the nucleotides. The sample is clarified with trichloroacetic acid and is then treated with a boiling mixture of ether and ethanol to remove lipids. The protein matter is then hydrolysed by heating at 37° for 24 hr. with 0·5N-NaOH, after which the clear soln. is adjusted to pH 4 to 4·5 with formic acid and the ppt. of proteins and deoxyribonucleic acids is separated by centrifugation. The clear liquid is passed through a column of cation-exchange resin (Dowex 50, H^+ form), from which the adsorbed ribonucleotides are eluted with water. The eluate is evaporated to small vol., and then submitted either to paper chromatography on Whatman No. 1 paper with phenol - isopropyl alcohol - formic acid - H_2O (17:1:2:20) as solvent system, followed by the micro-determination of nucleotide P, or to electrophoresis for 6 hr. on Whatman No. 1 paper at pH 3·5 (0·5M-citrate buffer, 10 V per cm) followed by u.v. spectrophotometric determination of the nucleotides. Both methods yield reasonably concordant results for the contents of guanylic, cytidylic, adenylic and uridylic acids in seven different tissues from the rat. W. J. BAKER

3436. **Detection of thyroid hormones in plasma by paper chromatography.** B. D'Alessandro, A. Brancaccio and G. Jacono (Ist. Semeiotic. Med., Univ., Napoli, Italy). *Biochim. Appl.*, 1960, **7** (6), 337–343.—The protein in 4 to 6 ml of plasma is pptd. with Somogyi's reagent (10% $ZnSO_4$ in 0·5N-NaOH). The washed ppt. is extracted first with methanol (10 ml) then with 70% methanol (6 ml). The combined extracts are evaporated to 4 ml. Water (2 ml) is added and the soln. is washed with light petroleum (4 × 6 ml). The washed soln. is evaporated to dryness at 40°, the residue re-dissolved in 0·3 ml of methanol containing 1% of conc. aq. NH_3 and subjected to two-dimensional ascending chromatography on Whatman 3MM paper. Standards of thyroxine and tri-iodothyronine are run on a second sheet. The solvent used for the first dimension is butanol - dioxan - 2N-aq. NH_3 (4:1:5) and, for the second dimension, butanol - 2N-formic acid (2:1), and the chromatograms are run for 12 to 13 hr. and 8 to 9 hr., respectively. The air-dried chromatogram is immersed in a mixture of 2% $Ce_2(SO_4)_3$ in N-H_2SO_4 - 0·2N-As_2O_3 - H_2O (1:1:1), dried and then immersed in 1% aniline in acetone and re-dried. The iodine compounds show up as white spots on a green ground and are identified by comparison with the standards. J. I. M. JONES

3437. **Method for the determination of urinary phenolic steroids.** A. Brancaccio, B. D'Alessandro and R. Repole (Ist. Semeiotic. Med., Univ., Napoli, Italy). *Biochim. Appl.*, 1960, **7** (6), 324–331.—Hydrolyse an aliquot of a 24-hr. sample of urine by boiling with HCl. Add NaCl, extract with ethyl ether, extract the ether extract with N-KOH, neutralise the alkaline extract and re-extract with ethyl ether. Evaporate the washed ether extract *in vacuo*, dissolve the residue in dichloromethane (**I**) and chromatograph on a Florisil column. Elute successively with 4% acetone in **I**, and 20% ethanol in **I**, collecting the eluates separately and evaporating to dryness. They contain, respectively, oestrone plus oestradiol, and oestriol. Dissolve each residue in ethanol, add quinol (2%) in H_2SO_4 and heat at 100° for 40 min. Cool, add *p*-nitrophenol in $CHCl_3$, shake for 20 min. and centrifuge. Measure the extinction of the $CHCl_3$ phase at 500, 538·5 and 577 mμ. Prepare two standards containing, respectively, oestrone (5 μg) plus oestradiol (5 μg), and oestriol (10 μg), and treat similarly with the reagent. Apply the Allen correction to the observed extinctions. J. I. M. JONES

3438. **Automatic recording of cholinesterase activities.** K. Jørgensen (Rigshospitalet, Copenhagen, Denmark). *Scand. J. Clin. Lab. Invest.*, 1959, **11** (3), 282–284.—A commercial automatic titration and recording apparatus is described, and its use in the determination of serum acetylcholinesterase is reported. D. B. PALMER

3439. **Colorimetric method for the estimation of monoamine oxidase.** A. L. Green and T. M. Haughton (Chem. Defence Exp. Estab., Porton Down, Wilts., England). *Biochem. J.*, 1961, **78** (1), 172–175.—This sensitive method is based on the measurement of the aldehyde formed during the enzymic oxidation of tyramine. The unstable aldehyde is prevented from decomposing by the presence of semicarbazide in the assay mixture. The semicarbazone so formed is then converted into the 2,4-dinitrophenylhydrazone, which is dissolved in aq. alkali. The deep-red colour of the soln. gradually changes to orange - yellow when the soln. is kept at room temp. for 1 hr. or at 80° for 10 min. This coloured form is stable in 0·1N-NaOH at room temp. for several hours, and determination of the extinction at 450 mμ gives a direct measure of the enzymic activity. Batches of 6 to 8 determinations can be carried out conveniently at one time. J. N. ASHLEY

3440. ***N*-Acetyl-β-glucosaminidase activity in serum during pregnancy.** P. G. Walker, M. E. Woollen and D. Pugh (Royal National Orthopaedic Hospital, Stanmore, Middlesex, England). *J. Clin. Path.*, 1960, **13** (4), 353–357.—A method for the determination of *N*-acetyl-β-glucosaminidase is described. Tubes containing 3·6mM-*p*-nitrophenyl *N*-acetyl-β-glucosaminide, 0·05M-citrate buffer (pH 4·3) and serum (0·1 ml) in a final volume of 1·0 ml are incubated at 37° for 30 min. The reaction is stopped and the liberated *p*-nitrophenol determined by measuring the extinction at 400 mμ after the addition of 0·25M-Na_2CO_3 (2·0 ml). The absorption of unhydrolysed substrate at this wavelength is negligible. After correction for blanks omitting serum or substrate the amount of *p*-nitrophenol liberated is read from a curve constructed with a standard soln. of *p*-nitrophenol. One unit of the enzyme is defined as that quantity that will liberate 1 mg of *p*-nitrophenol under these conditions. R. A. BRENAN

3441. **Studies on glucosaminidase. IV. The fluorimetric assay of *N*-acetyl-β-glucosaminidase.** D. H. Leaback and P. G. Walker (Biochem. Dept., Inst. of Orthopaedics, Royal Nat. Orthopaedic Hosp., Stanmore, Middx., England). *Biochem. J.*, 1961, **78** (1), 151–156.—The enzyme soln. is incubated at 37° for 30 min. with 0·05M-Na citrate buffer (pH 5·0), 0·01% of albumin and 0·264mM-methylumbelliferyl *N*-acetyl-β-glucosaminide. The enzymic reaction is then stopped and 0·2M-Na glycinate buffer (pH 10·65) is added. The methylumbelliferone which has been liberated is then determined spectrophotometrically at 360 mμ (for the range of 0·5 to 11 μg per ml) or fluorimetrically (for the ranges 0·01 to 0·45 μg per ml or 0·0025 to 0·10 μg per ml). The actual amount of enzyme is then ascertained from standard graphs. The range and sensitivity of these methods are superior to those of existing methods. J. N. ASHLEY

3442. **Studies on elastase and elastin. I. Assay and properties of elastase.** F. Lamy, C. P. Craig and S. Tauber (Dept. of Anat., Sch. of Med., Univ. Pittsburgh, Pa., U.S.A.). *J. Biol. Chem.*, 1961, **236** (1), 86–91.—Three assay methods are described. The first is based on the determination of the sol. polypeptides liberated by the action of elastase on elastin, the second depends on the titrimetric determination of the carbonyl groups liberated during hydrolysis of the peptide bonds in the substrate, and the third is based on the use of ninhydrin reagent for the colorimetric determination of the free amino-groups released during the cleavage of the peptide bonds. This third method is approx. three times as sensitive as the second and five times as sensitive as the first. J. N. ASHLEY

See also Abstracts—**3191**, ^{90}Sr in biol. materials. **3347**, 2,4-Diaminophenol in urine. **3363**, Amide N in gelatin. **3484**, Vitamin D in animal tissue. **3499**, Sugar and starch in vines. **3509**, Chromatography of amino-acids and peptides. **3547**, pH of blood.

Pharmaceutical analysis

3443. **Application of column chromatography to the analysis of drugs.** J. A. P. Stroes (Pharm. Lab., Rijksuniv., Groningen, The Netherlands). *Pharm. Weekbl.*, 1961, **96** (2), 49–56.—The application of ion exchangers and column partition chromatography to the analysis of mixtures of drugs was investigated. A mixture of allylbarbituric acid, phenazone and caffeine can be analysed with the aid of the strongly acid resin Dowex 50-X2 washed with 15% HCl and then with H_2O. Caffeine and the barbituric acid derivative are eluted with ethanol and determined in the eluate by u.v. spectrophotometry. Phenazone is eluted with N-aq. NH_3 and subsequently determined by u.v. spectrophotometry. A mixture of acetylsalicylic acid, caffeine citrate, ascorbic acid and ephedrine hydrochloride can be analysed by using Amberlite IRA-400 washed with N-NaOH and then with H_2O. The two acids are retained by the resin, whereas caffeine runs through and is determined in the percolate by u.v. spectrophotometry. Ephedrine does not interfere in this determination. The following mixtures were analysed by column partition chromatography, by using Celite 545—(*i*) acetylsalicylic acid, phenacetin, caffeine and colloidal $Al(OH)_3$ and (*ii*) barbituric acid and aprobarbital. M. J. MAURICE

3444. **Assay of pharmaceutical substances in suppositories by titration with perchloric acid in acetic acid.** M. Hädicke and K. Howorka (Staat. Inst. f. Arzneimittelprüfung, Jena, Germany). *Pharm. Zentralh.*, 1961, **100** (1), 3–5.—The determination of morphine hydrochloride, papaverine hydrochloride, amidopyrine and Spasmonal (β-piperidinoethyl α-phenyl-α-piperidinoacetate dihydrochloride) is described. If the suppository base is a water-soluble polyoxyethylene glycol, the water is removed by heating to 105° or by addition of acetic anhydride before the assay. Barbituric acid and phenacetin do not interfere. A. R. ROGERS

3445. **Use of formaldehyde-treated alginic acid in the chromatographic determination of organic bases.** J. S. Foster and J. W. Murfin (Standards Dept., Boots Pure Drug Co. Ltd., Airdrie, Scotland). *Analyst*, 1961, **86**, 32–36.—Alginic acid, after suitable treatment with formaldehyde, can be used as a carboxylic cation-exchange medium for quant. separation of organic bases from solution. The alginic acid is mixed to a damp paste with formaldehyde soln. (40%), heated in a loosely stoppered jar at 80° for 8 hr. and then dried at 100°. The broken-up material is sifted through 22- and 100-mesh sieves, the granular powder remaining between the sieves being retained for use. To prepare the column, a slurry of the prepared alginic acid with 10% (w/w) H_2SO_4 is placed in a plugged tube and the column is washed with N-H_2SO_4 until the percolate shows no measurable extinction between 220 and 350 mμ, and then with water until free from acid. Applications to the adsorption and subsequent determination of 14 organic bases (*e.g.*, alkaloidal salts and adrenaline) and to determinations of codeine phosphate in Compound Tablets of Codeine B.P. and strychnine in Prepared Nux Vomica B.P.C., 1954, and Tincture of Nux Vomica B.P. are described. A. O. JONES

3446. **Problems of the [German] Pharmacopoeia. New method for determination of alkaloid content of drugs.** H. Auterhoff and C. Becker (Inst. f. pharm. Chemie, Tech. Hochschule, Braunschweig, Germany). *Arch. Pharm., Berlin*, 1960, **293**, 1021–1029.—Current official methods are considered inadequate for dealing with some drugs. The following method gives results for the alkaloids (as emetine) in ipecacuanha, 5 to 6% higher than those obtained by the D.A.B. VI method, and in good agreement with those obtained by the procedure given in improved methods of the D.A.B.; the new method is somewhat more accurate and less laborious than the latter procedure. *Procedure*—Boil the powdered sample (2 g) for 5 min. with three successive portions (20, 15, 10 ml) of anhyd. acetic acid (free from acetic anhydride), make the combined filtered extracts alkaline with aq. NH_3 soln., and then extract the alkaloids with $CHCl_3$ (100, 50, 50 ml). Dry the combined extracts with Na_2SO_4, and evaporate to dryness *in vacuo*. Dissolve the residue in anhyd. acetic acid (10 ml) and titrate with 0·1N-$HClO_4$ in anhyd. acetic acid, with crystal violet as indicator; [for titrating coloured soln., as obtained from cinchona bark, the indicator Fettblau B (C.I. Solvent Blue 19) gives the best results]. The presence of H_2O (0·4%) incurred by the use of 70% $HClO_4$ increases the results by 0·7%. Shorter boiling times with acetic acid (1 to 2 min.) may be used for cinchona bark or belladonna leaves. P. S. ARUP

3447. **Photometric determination of alkaloids in non-aqueous solution.** I. Bayer (Pharmakopöelab. des Min. f. Gesundheitswesen, Budapest, Hungary). *Pharm. Zentralh.*, 1961, **100** (1), 6–12.—A simple method is described for the determination of salts of alkali metals, alkaloids and their salts, and compounds such as amidopyrine, oxytetracycline and proguanil; barbiturates, caffeine, phenacetin, acetylsalicylic acid and purines do not interfere. *Procedure*—Prepare a soln. of the sample (≃50 mg) in acetic acid (100 ml) and take a 2-ml aliquot. If the sample contains halide ions, add a 0·1% soln. of mercuric acetate in acetic acid (2 ml). Dilute to 5 ml with acetic acid, mix with a 1% soln. of picric acid in acetic acid (5 ml) and compare the extinction in a Pulfrich photometer with that of a 0·5% soln. of picric acid, in 2-cm cells with filter S43. The acetic acid used as solvent must contain <1% of H_2O. The coloured soln. obeys Beer's law.
A. R. ROGERS

3448. **Countercurrent partition to separate and determine the alkaloids of *Hydrastis canadensis*.** G. Biglino (Univ. Turin, Italy). *Boll. Chim. Farm.*, 1960, **99**, 195–200.—Countercurrent partition makes it possible to separate hydrastine, canadine and berberine in artificial mixtures as well as in tinctures of *H. canadensis*. With a 200-tube apparatus the best pairs of solvents were t-amyl alcohol - borate buffer (pH 8) and t-butyl alcohol - phosphate buffer (pH 7). The assay in extract and tinctures is preceded by percolation through an alumina column to eliminate inert coloured substances, followed by elution with 85% ethanol. Berberine is determined spectrophotometrically in the eluate at 348 mμ. The eluate is then concentrated, and hydrastine and canadine are extracted with ethyl ether after adding aq. NH_3, and separated by countercurrent partition between aq. benzene and a mixture of 2 litres of 95% ethanol and 650 ml of H_2O by dissolution in the ethanolic phase. For spectrophotometry the liquids are made up to vol. with dioxan. The wavelengths used are 285 mμ for canadine and 297 mμ for hydrastine.
CHEM. ABSTR.

3449. **The control of injections and other pharmaceutical preparations by means of u.v. spectrophotometry as a standard analytical tool. VI. Spectrophotometric assay of atropine sulphate, pethidine hydrochloride and methadone hydrochloride.** H. J. van der Pol and R. F. Rekker (N.V. Kon. Pharm. Fabr. v/h Brocades-Stheemann en Pharmacia, Amsterdam-Meppel, The Netherlands). *Pharm. Weekbl.*, 1961, **96** (2), 41–48 (in English).—Some additional figures on the spectra of atropine sulphate, obtained with a recording spectrophotometer Unicam SP 700, are given (*cf. Anal. Abstr.*, 1960, **7**, 3947), and the spectrophotometric determination of pethidine hydrochloride (at 257 mμ) in injections and tablets, and methadone hydrochloride (at 292·5 mμ) in injections is described. The spectrophotometric determination of methadone hydrochloride in tablets has not yet proved satisfactory.
M. J. MAURICE

3450. **Chromatographic separation on paper of colchicine and colchamine.** V. M. Slavo. *Med. Prom. SSSR*, 1960, (4), 39; *Ref. Zhur., Khim., Biol. Khim.*, 1960, (21), Abstr. No. 29,492.—A mixture of 0·1% methanolic colchicine soln. (**I**) and 0·11% methanolic colchamine soln. (**II**) is put on to paper (which has been placed for 1 to 2 hr. in a chamber satd. with steam) and chromatographed with H_2O by the ascending method; **I** zones give a blue fluorescence in u.v. light and **II** zones give a green - blue fluorescence; the R_F value for **I** is 0·7 to 0·8 and that for **II** is 0·4 to 0·5. **I** can also be located by 1% aq. molybdophosphoric acid and **II** by Dragendorff's reagent.
K. R. COOK

3451. **Chemical evaluation of digitalis drugs (*D. purpurea* and *D. lanata*). IV.** W. Hauser and W. Berger (Pharmakog. Inst., Univ. Graz, Austria). *Sci. Pharm.*, 1961, **29** (1), 39–44 (in German).—The procedure described previously for the determination of the "value number" (*cf. Anal. Abstr.*, 1961, **8**, 776) has been more closely defined. An acceptable sample of the drug has a "value number" in the range 9 to 15. *Procedure*—Macerate the drug in medium powder with H_2O for 1 hr. with agitation, filter and extract the filtrate with $CHCl_3$. Apply a portion of the extract to a column of alumina previously treated with a 2·5% soln. of formic acid in ethanol, elute digitoxin and gitaloxin with a 4% soln. of 96% ethanol in $CHCl_3$, and determine by the method of Kedde.
A. R. ROGERS

3452. **Quantitative determination of rutin and aesculin in a drug mixture by chromatographic separation on polyamide powder.** L. Hörhammer, H. Wagner and O. Föcking (Inst. Pharm. Arzneimittel, Univ., Munich, Germany). *Arch. Pharm., Berlin*, 1960, **293**, 1001–1005.—Soln. of mixtures of the pure compounds (3 mg) are passed through a column of the powder [4 g, previously mixed with water and washed with water (50 ml)]; the aesculin (**I**) is eluted with water, and then the rutin (**II**) with methanol, both with 100% recovery. Separate graphs are given for mg of **I** and **II** per 10 ml against extinctions at 375 and 360 mμ in their respective soln. (The extinction peak for **I** is shifted from 335 to 375 mμ on passage through the column, but partial reversion is observed after 1 hr.) From a prep. containing theophylline, ascorbic acid and a salt of Mg in addition to **I** and **II**, recoveries of **I** were ≃110% for sterilised, and 100% for unsterilised, soln.; for **II** they were 72 to 73% in both cases. Changes probably undergone by **I** and **II** on passage through the column are considered.
P. S. ARUP

3453. **Quantitative differentiation between natural capsaicin (8-methyl-*N*-vanillylnon-6-enamide) and [the synthetic compound] *N*-vanillylnonanamide.** P. R. Datta and H. Susi (U.S. Dept. Agric., Philadelphia, Pa.). *Anal. Chem.*, 1961, **33** (1), 148.—The method is based on the i.r. absorption of natural capsaicin at 970 cm^{-1}; *N*-vanillynonanamide does not absorb at this wavelength. Both compounds absorb equally at 1040 cm^{-1} and therefore can be individually determined.
G. P. COOK

3454. **Chemical method for determination of cycloserine.** J. Kolesińska (Inst. of Antibiotics, Warsaw, Poland). *Chem. Anal., Warsaw*, 1960, **5** (6), 1033–1037.—*Procedure*—The test sample is diluted (1:9) with 0·1N-NaOH. To 1 ml of this soln. are added N-acetic acid (3 ml) and reagent [4N-NaOH - 4% aq. sodium nitroprusside soln. (1:1)] (1 ml). After 10 min. the extinction is measured at 610 mμ and compared with a calibration graph, which is linear over the range 5 to 150 μg of cycloserine per ml. The results obtained by this method agree with those obtained by biological assay.
B. K.

3455. **Spectrophotometric determination of chloramphenicol and its esters in the presence of sulphonamides.** F. Lorenz and G. Machek (Kontroll-Lab. der "Biochemie" G.m.b.H., Kundl/Tirol). *Sci. Pharm.*, 1961, **29** (1), 12–19 (in German).—The method is based on measurement of the extinction at 270 mμ of an ethanolic soln. of the sample (containing ≃1 μg of chloramphenicol per ml) and subtraction of a correction for the contribution due to the content of sulphonamides determined by the Bratton - Marshall method. The relative error is ≃1% for samples containing 10 to 50% of chloramphenicol, and ≃5% for samples containing 1 to 2%. A. R. ROGERS

3456. **Detection of some analgesics and antipyretics with paper chromatography.** J. Turek and B. Chundela (Lab. Toxicol. and Forensic Med., Charles' Univ., Prague). *Českosl. Farm.*, 1961, **10** (1), 28–29.—Phenazone, phenacetin and caffeine, which are extracted into $CHCl_3$ from an acid medium during the Stas - Otto procedure, can be chromatographed on Whatman No. 1 paper with light petroleum - butanol - H_2O (10:3:5) as solvent and the descending technique. By photographing the spots in u.v. light at 254 mμ, as little as 10 μg of the substance can be detected. J. ŽÝKA

3457. **Determination of benzocaine in mixtures with procaine.** G. Di Bacco (Univ. Rome, Italy). *Boll. Chim. Farm.*, 1960, **99**, 208–211.—A soln. of 250 mg of the sample is made up to 50 ml with H_2O. An aliquot (2 ml) is mixed with H_2O (3 ml), 25% HCl (10 drops) and enough 2·5% reinecke salt soln. to produce a pink colour. The mixture is centrifuged, the supernatant liquid is removed, H_2O is added to make up to 10 ml, the mixture is centrifuged again, and the washing is repeated. The precipitate is dissolved in acetone, made up to 25 ml and compared colorimetrically with a standard of procaine. An aliquot (40 ml) of the original soln. and 10 ml of HCl is titrated in the cold with 0·1M-$NaNO_2$ (1 ml ≡ 2·73 mg of procaine and 1·65 mg of benzocaine). The benzocaine is obtained by difference. CHEM. ABSTR.

3458. **Polarography of iodochlorhydroxyquin and di-iodohydroxyquin.** L. W. Brown and E. Krupski (Coll. of Pharm., Univ. of Washington, Seattle, U.S.A.). *J. Pharm. Sci.*, 1961, **50** (1), 49–51.—5-Chloro-8-hydroxy-7-iodoquinoline (iodochlorhydroxyquin) (**I**) and 8-hydroxy-5,7-di-iodoquinoline (di-iodohydroxyquin) (**II**) may be determined polarographically in 10^{-3}M soln. with an accuracy of ±2%. Use a soln. of 0·024M-K acetate, 0·013M-acetic acid and 0·06M-LiCl in 90% ethanol, adjusted to pH 7, as solvent for **I**, and a soln. of 0·05M-tetraethylammonium bromide in 2-ethoxyethanol as solvent for **II**. The $E_{\frac{1}{2}}$ *vs.* the S.C.E. are, for **I**, −1·3 V, and, for **II**, −1·5 V. A. R. ROGERS

3459. **Properties and analysis of dextromoramide and its dosage forms.** P. J. A. W. Demoen (Res. Lab. Dr. J. Janssen, Beerse, Belgium). *J. Pharm. Sci.*, 1961, **50** (1), 79–82.—Some analytical characteristics of dextromoramide (**I**) and its tartrate are given. In the absence of interfering substances, **I** may be determined by titration in glacial acetic acid with $HClO_4$ or by measurement of the u.v. absorption at 261·5 mμ in isopropyl alcohol. To determine **I** in tablets containing interfering substances, extract the powdered sample with 1% tartaric acid soln., shake with ether and discard the ethereal extracts; then render the aq. phase alkaline, extract **I** with $CHCl_3$, evaporate to dryness and determine **I** spectrophotometrically or by non-aqueous titration. A. R. ROGERS

3460. **Colorimetric determination of benactyzine in pharmaceutical preparations.** W. Chiti (Lab. di Ric. della "Ibis," Firenze, Italy). *Farmaco, Ed. Prat.*, 1960, **15** (12), 759–763.—Benactyzine (**I**) is pptd. quant. from aq. soln. by molybdophosphoric acid, and the ppt. dissolves in acetone to give a green soln. *Procedure*—Dissolve the powdered sample (10 to 50 mg of **I**) in H_2O (30 ml) containing HCl (1:2) (0·5 ml) and heat for 5 min. on a boiling-water bath. Filter and wash the residue with hot water (10 and 5 ml). To the cold filtrate and washings add $NaHCO_3$ (1 g) and when this has dissolved extract with ethyl ether (3 × 20 ml). Wash the extract with H_2O (10 ml), then extract thrice with HCl (1:20) (total vol. 30 ml). To the acid extract add, dropwise, fresh 20% molybdophosphoric acid soln. (4 ml). Filter off the ppt., wash it with 10% HCl (3 × 5 ml), dry it by suction, then dissolve it in acetone (3 × 5 ml). Dilute the soln. to 50 ml with acetone, and measure the extinction at 400 mμ (for ≃10 mg of **I**) or 440 mμ (for ≃50 mg of **I**). Prepare standards from a soln. of **I** (10 mg per ml) in 5% HCl. The method is accurate to within 2%. J. I. M. JONES

3461. **Determination of selenium in prednisone and its derivatives.** K. Heller and U. Wagner (Wissenschaftl. Lab., VEB Jenapharm, Jena, Germany). *Z. anal. Chem.*, 1961, **178** (6), 428–431 (in German).—Commercial preparations of prednisone may contain 0·2 to 0·6% of Se, which may be determined by combustion of the sample in O and iodimetric titration of the selenous acid formed. T. R. ANDREW

3462. **Paper-chromatographic identification and determination of polyoxyethylene glycol dodecyl ether.** D. Pertsemlides and K. Soehring (Pharmakol. Inst., Univ., Hamburg). *Arzneimittel-Forsch.*, 1960, **10** (12), 990–996.—*Identification*—Polyoxyethylene glycol dodecyl ether (DoR9) (**I**) is separated from amethocaine, procaine, lignocaine and Carbocaine (*N*-methylpipecolic acid 2,6-xylidide) in aq. soln. by ascending paper chromatography with n-butanol - acetic acid - H_2O (4:1:5) and detected by Dragendorff's reagent; 10 μg of **I** can be detected. **I** in blood or urine is first extracted into CCl_4; 5 μg of **I** per ml can then be detected. *Determination of* **I** *with* $KMnO_4$—Mix the soln. (5 to 20 ml, containing 100 to 400 mg of **I**) with 10% $K_4Fe(CN)_6$ soln. (10 ml) and H_2SO_4 (2 ml), dilute to 50 ml and filter. Dilute an aliquot of the filtrate (25 ml) with H_2O (100 ml) and titrate the excess of $K_4Fe(CN)_6$ with 0·1N-$KMnO_4$. *Photometric determination of* **I**—Mix the aq. test soln. (10 ml) with N-HCl (10 ml) and 0·001N-iodine (10 ml), dilute to 100 ml and measure the extinction against a blank at 295 mμ (accuracy ±5%) or 375 mμ (accuracy ±7·4%), or measure the extinction with a Hg-366 filter. A. G. COOPER

3463. **Assay of zinc in soft medicinal preparations.** G. Lukas (Abt. für galen. Pharm., Inst. für organ. und pharm. Chem., Univ. Graz, Austria). *Sci. Pharm.*, 1961, **29** (1), 24–31 (in German).—Gelatin of zinc and certain pastes and ointments containing ZnO can be assayed by burning the sample (≃100 mg) wrapped in filter-paper in a closed flask containing O (500 to 1000 ml) according to the technique of Schöniger, followed by absorption of the

products of combustion in 2N-HNO_3, dilution with H_2O and titration with 0·1M- or 0·02M-EDTA (disodium salt), with Eriochrome black T as indicator. High results may be obtained with preparations containing certain grades of talc.
A. R. ROGERS

3464. **Application of gas chromatography in toxicological analysis. Poisoning by paraldehyde and turpentine oil.** G. Machata (Inst. f. gerichtl. Med., Univ., Wien). *Archiv. Toxikol.*, 1960, **18**, 338–346.—For paraldehyde, steam-distil the stomach contents or the pulverised lung or brain tissue and extract the distillate with ether; evaporate the ether and submit a small portion to chromatography on silicone oil at 100° with N as carrier gas. For turpentine oil, steam-distil the acidified contents of the intestine and extract the distillate with pentane; evaporate the solvent and submit a small portion to gas chromatography on silicone oil at 130° or on polyoxyethylene glycol at 75° with N as carrier gas. Identification is confirmed by infra-red spectroscopy.
A. R. ROGERS

3465. **Physico-chemical analyses in poisoning by Thorotrast.** G. Machata (Inst. f. gerichtl. Med., Univ., Wien). *Beitr. gerichtl. Med.*, **21**, 34–39.—Emission spectrography and γ-ray spectrometry have been used to identify and determine as little as 1 mg of Th (from the use of Thorotrast as an X-ray contrast medium) in the ash of biological material. In cases of poisoning, Th is concentrated mainly in the liver, the spleen and the lymph nodes.
A. R. ROGERS

See also Abstracts—**3352**, Identification of pyrimidine deriv. **3406**, Sulphaphenazole in biol. material. **3407**, N^4-acyl sulphonamides in animal tissues.

Food

Foods and food additives, beverages, edible oils and fats, vitamins.

3466. **Determination of aldehydes and ketones in foodstuffs. II. Titrimetric micro-determination of unsaturated aldehydes, ketones and other unsaturated compounds by means of bromide - bromate solution.** V. Hamann and A. Herrmann (Dtsch. Forschungsanst. für Lebensmittelchem., München, Germany). *Mikrochim. Acta*, 1961, (1), 105–128 (in German).—A method for the quant. isolation of volatile oils by continuous steam-distillation is described; recoveries for citral were about 92%. The determination of volatile oils, terpenes, unsaturated hydrocarbons, alcohols and acids by reaction with KBr - $KBrO_3$ soln. and back-titration of Br was studied, and recoveries are tabulated. For unsaturated hydrocarbons such as octene, Hg^{II} acetate was used as a catalyst.
P. D. PARR-RICHARD

3467. **Ion-exchange method for determining cations and the ash content of sugar products.** S. Zagrodzki and H. Zaorska (Polytech., Łódź, Poland). *Roczn. Technol. i Chem. Żywności*, 1960, **5**, 5–15.—This method is used for the determination of K, Na, Ca and Mg, the main components of sugar ash. The sample soln. is passed through a column filled with a polystyrene cationite containing active SO_3H groups, *e.g.*, Wofatit KPS-200 resin, treated with aq. NH_3. With viscous soln., the operation is carried out at 60° or 80°, maintained by means of a hot-water jacket. The column is then washed with water and the washings are discarded. The K^+ and Na^+ are eluted with 2N-ammonium carbonate and this first eluate is set aside. The resin is then regenerated with 3·5N-HCl and washed with water, and the Ca^{2+} and Mg^{2+} are eluted with 25% aq. NH_3 followed by water. The sum of K^+ and Na^+ is determined in the first eluate, after removal of the excess of ammonium carbonate, by titration with 0·5N- or 0·2N-HCl to methyl red. The sum of Ca^{2+} and Mg^{2+} is determined by titrating a known amount of EDTA (disodium salt) soln., buffered with aq. NH_3, with the second eluate, with Eriochrome black T as indicator. The "carbonate ash" or "sulphate ash" can be calculated from these titrations. When larger amounts of Fe, Al or other cations are present in the sample, these can be individually determined in the second eluate. The method takes only 90 min., and the ion-exchange column is ready for the next determination after the second elution.
A. L. GROCHOWSKI

3468. **Determination of proteins in whole wheatmeal and flour by the biuret procedure.** P. C. Williams (Agric. Res. Inst., Wagga Wagga, N. S. Wales, Australia). *J. Sci. Food Agric.*, 1961, **12** (1), 58–61.—The biuret reaction is adapted for a colorimetric determination of protein over the range 6 to 19% in wheatmeal and 5·8 to 17·6% in flour. Lignified materials may interfere. *Procedure*—To flour or finely ground wheatmeal (5 g) add CCl_4 (1 ml) and biuret reagent (50 ml) in a stoppered flask and shake for 10 min. Set aside for 1 hr. and centrifuge 10 to 20 ml of the suspension at 3000 r.p.m. for 5 min. Compare the colour of the supernatant liquid with a reagent blank in 2-cm cells, with an Ilford 605 filter, and calculate protein from a graph of Kjeldahl protein *vs.* biuret protein.
M. A. ELLIS

3469. **Moisture determination in baked goods and doughs by automatic titration with Karl Fischer reagent.** P. F. Pelshenke, W. Seibel and H. Bolling (Bundesforschungsanst. für Getreideverarbeit., Detmold, Germany). *Dtsch. Lebensmitt-Rdsch.*, 1960, **56** (10), 293–296.—The application of automatic titration by the dead-stop end-point technique with Karl Fischer reagent to the rapid determination of moisture in doughs and freshly baked goods is described. Results obtained are somewhat higher than by oven-drying methods, but these differences are probably due to incomplete removal of moisture in normal drying.
E. C. APLING

3470. **Synthetic carotenoids for colouring foodstuffs.** O. Isler, R. Rüegg and P. Schudel (F. Hoffman-La Roche & Co. A.-G., Basel). *Chimia*, 1961, **15** (1), 208–226 (in German).—Synthetic carotenoids used as colouring agents in foodstuffs are separated by chromatography on activated alumina and elution with ethyl ether - light petroleum. The eluted β-carotene can be quant. determined by visible and infra-red absorption spectra. Structural analysis is carried out via nuclear magnetic resonance and X-ray powder techniques. Applications of these techniques to the β-apocarotenal series are given.
G. P. MITCHELL

3471. **Electrophoretic determination of synthetic dyes permitted as additives in Italy.** M. T. Guzzoni (Ist. Chim. Farm., Univ. Pavia, Italy). *Farmaco, Ed. Prat.*, 1960, **15** (12), 752–758.—Permitted synthetic colouring matters for use in foods in Italy are characterised and identified by electrophoresis on S. & S. 2043b paper in a buffer soln. of pH 11 and

ionic strength 0·01, at 250 V, for 6 hr. A table is given of the coloured bands and their displacements under these conditions for the 13 permitted dyes. Sugars do not affect the displacements. Foodstuffs are dissolved or suspended in H_2O (25 ml) and extracted with n-butanol (3, 3, 2 and 2 ml), and the extract (0·1 ml) is subjected to electrophoresis; quant. determination is effected by elution of the bands and colorimetric measurement.

J. I. M. JONES

3472. **Quantitative determination of *p*-hydroxybenzoic acid in cheese.** F. Kiermeier and R. Jarczynski (Chem. und Phys. Inst., Süddeutschen Versuchs- und Forschungsanst. für Milchwirtschaft, Weihenstephan). *Z. LebensmittUntersuch.*, 1960, **113** (5), 370–374.—To determine traces of naturally produced *p*-hydroxybenzoic acid (**I**) in cheese, 50 g of cheese freed from crust is boiled under reflux for 30 min. with 100 ml of 26% HCl, and the hot mixture is transferred to a wet filter and washed with a minimum amount of hot water. After cooling, the filtrate is extracted (×4) with a total of 300 ml of ether. The ether, after being dried with anhyd. Na_2SO_4, is distilled off in a flask that has a small graduated tube sealed on to the bottom to collect the residue. The residue in this tube is dissolved in acetone to a vol. of 2·0 ml, and aliquots (10 to 50 µl) are chromatographed by the descending technique on S. & S. No. 2043b mgl paper, previously soaked in 0·2M-Na_2CO_3 and dried, with butanol - water (3:1) as mobile phase. After allowing 1 hr. for saturation in the tank atmosphere, the paper is developed for 14 hr., the moving front travelling ≃ 35 cm. Spots from control aliquots on the outer strips of the paper are treated with diazotised sulphanilic acid (0·1 g in 20 ml of 10% Na_2CO_3 soln.) to locate the **I**, which appears near the starting line (R_F = 0·06). The located spot on the middle strip of paper is cut out, placed in a dry 25-ml flask with 2 ml of aq. 4% $Na_2B_4O_7$ and shaken at intervals for 20 min. A fresh soln. of diazotised *p*-nitroaniline is prepared (method described), of which 1·0 ml is added to the eluted **I**. After 15 min. the vol. is made up with water, paper fibres (which interfere with the determination) are removed by filtration, and after 30 min. the colour of the soln. is measured with filter S47 and a 5-cm cell. For the calibration curve, aliquots of a standard soln. of **I** in acetone must be treated as for the cheese. Amounts of **I** found in soft cheese were ≥ 0·4 mg per 100 g.

C. L. HINTON

3473. **Determination of the ammonia content of tea.** V. P. Popov. *Biokhimiya Chaĭn. Proiz. 7 M. Akad. Nauk SSR*, 1959, 182–188; *Ref. Zhur., Khim., Biol. Khim.*, 1960, (21), Abstr. No. 29,495.—The usual methods employed in the determination of NH_3 in tea and other vegetable matter with a high tannin content yield misleading results because of the formation of NH_3 during the analytical procedure; thus, in the presence of alkali, tannins are oxidised to quinone which contributes to the oxidative deamination of amino-acids, resulting in the liberation of NH_3. The amount of NH_3 formed depends on the type and concn. of polyphenols or amino-acids present. If ascorbic acid, which prevents the oxidation of tannins, is added, a correct figure for the NH_3 content can be obtained. It is proved that the formation of NH_3 during tea fermentation arises from the oxidative deamination of amino-acids by quinones, which are produced by the oxidation of tannins by polyphenoloxidase.

K. R. COOK

3474. **Determination of riboflavine in barley, malt and yeast.** L. Maloia. *Birra e Malto*, 1960, (Aug.), 52–53.—Two samples of each material (ground for barley and malt), each containing ≃ 100 µg of riboflavine, were suspended in 150 ml of water, one sample being fortified with 100 µg of riboflavine added as 1 ml of a standard soln. After shaking the suspensions for 10 min. in boiling water, they were cooled to 45°, 0·5 g of Clarase was added and the mixtures were incubated at 45° for 20 min. The digests were cooled to 20° to 25°, 0·5 ml of glacial acetic acid was added and the mixtures were made up to 250 ml. Portions (10 ml) were withdrawn and extracted with $CHCl_3$ (60 ml) to remove interfering fluorescent substances, and the resulting emulsions were centrifuged. The supernatant liquids were treated with 25 ml of KOH soln. (250 g in 750 ml of water) and each ppt. was centrifuged. Portions (10 ml) of the supernatant liquids were irradiated in shallow layers for 20 min. at a distance of 20 cm from a mercury-vapour lamp, after which the soln. were acidified with 2 ml of glacial acetic acid, shaken with $CHCl_3$ (15 ml) and centrifuged. From the $CHCl_3$ layer, 10-ml portions were taken out and made up to 25 ml with ethanol. From the measured fluorescence of these soln. the riboflavine contents of the samples were calculated.

J. INST. BREWING ABSTR.

3475. **Application of formazin standards to nephelometric estimation of beer turbidity.** R. S. W. Thorne (A. Jørgensen Lab. for Fermentation, Copenhagen). *J. Inst. Brewing*, 1961, **67** (2), 191–199.—The theory and practice of nephelometry are discussed. When the conditions of use are strictly specified, the E.B.C. formazin haze unit (*cf.* Bishop, *Anal. Abstr.*, 1961, **8**, 1723) has a definite and reproducible absolute turbidity, and can be used for the calibration of conventional nephelometers, which, owing to the complexity of the light-scattering phenomenon, cannot measure true absolute turbidities. Nephelometers of different design will each distort the unit in its own way, and will therefore yield results that are inconsistent with each other and with the true turbidity. Formazin standards will therefore yield an arbitrary scale for individual instruments, but not an absolute one. This is a defect, not of the standard itself, but of practical nephelometry; any haze standard will be subject to similar limitations.

R. E. E.

3476. **Polyhydric alcohols in wine. Chromatographic method for detecting the addition of cider.** E. Emiliani and I. Ucha de Davie (Inst. de Invest., Dept. de Microbiol., Univ. Nac. del Litoral, Santa Fé). *Rev. Fac. Ing. Quím., Argentina*, 1959, **28**, 93–104.—The method depends on the separation and determination of sorbitol, of which Argentinian wines contain 2 to 6 mg per 100 ml, whereas cider contains 0·4 to 1%. *Procedure*—The sample (50 ml) is heated on a boiling-water bath for 15 min. with activated charcoal (2·5 g) and filtered; an aliquot (10 ml) of the filtrate is evaporated to dryness, the residue is dissolved in water (0·25 ml) and transferred to Whatman No. 1 paper. The chromatogram is developed by the ascending technique for 10 to 12 hr. with ethyl acetate - 95% ethanol - water - glacial acetic acid (65:20:15:1) and dried in air. The lower part of the paper, containing interfering substances, is cut off and the upper part of the paper is immersed in a suspension of ammonium molybdate in acetone and dried in air. The chromatogram is then re-developed for 5 to 7 hr.

with butanol - pyridine - water (43:42:15). The spots are revealed by treatment with ethanolic NaOH soln.; the sorbitol - molybdate complex has a low R_F value. The determination is made by comparison with a standard of sorbitol. The addition of 2% of cider to wine can be detected.
R. E. E.

3477. **Polarographic determination of L-ascorbic acid in wine.** W. Diemair, J. Koch and D. Hess (Univ. Inst. f. Lebensmittelchem., Frankfurt/Main, Germany). *Z. anal. Chem.*, 1961, **178** (5), 330–335 (in German).—The sample (30 ml) with 15 ml of de-aerated acetate buffer soln. at pH 5·33 (prep. described) is transferred to a polarographic cell at 20° and determined polarographically by using an anodic polarised dropping-mercury electrode from −0·1 to +0·25 V. Oxygen and free H_2SO_3 interfere; the former is removed with a stream of O-free N and the latter with acetaldehyde, which is added to the buffer soln. The diffusion current is proportional to the concn. of ascorbic acid and the measured values are referred to a calibration curve. The results are compared with those by an iodimetric method and by the method of Müller (*Dtsch. Weinztg*, 1955, **37**, 314). B. B. BAUMINGER

3478. **Detection and determination of thujone in wine.** W. Diemair and G. Weinberger (Univ.- Inst. f. Lebensmittelchem., Frankfurt/Main, Germany). *Z. LebensmittUntersuch.*, 1960, **113** (5), 374–380.—Cases of illness due to consumption of vermouth with a high content of volatile-oil constituents, chiefly thujone, led to an examination of the method of Joppien (*Anal. Abstr.*, 1957, **4**, 1889) for the determination of thujone after conversion into the 2,4-dinitrophenylhydrazone. No significant differences were found between the transmission curves of wine with added thujone and those of untreated wine, nor did the transmission minimum of 360 mμ due to the thujone afford clearly reproducible results. Descending chromatographic separation gave rather better results, but only when large amounts of thujone are present can transmission and extinction values afford a more or less certain indication of its presence. C. L. HINTON

3479. **Determination of total and free sulphurous acid in wine.** W. Diemair, J. Koch and D. Hess (Univ. Inst. f. Lebensmittelchem., Frankfurt/Main, Germany). *Z. anal. Chem.*, 1961, **178** (5), 321–330 (in German).—Various methods are discussed for the determination of total H_2SO_3, and the method proposed differs from that of Deibner and Bénard (*Ind. Agric. Aliment.*, 1953, **70**, 11) in the use of citric acid - phosphate buffer (**I**) at pH 3·4 as absorbing soln. The sample (25 ml) is mixed with 5 ml of de-aerated H_3PO_4 (85%) and 75 ml of de-aerated H_2O and distilled (apparatus described) into a receiver containing 10 ml of **I**. To the distillate is added 1 ml of starch soln. (1%) and the soln. is titrated with 0·01N-iodine. Near the end-point 1 g of $NaHCO_3$ is added and titration is continued. The results are corrected by a reagent blank. For the determination of free H_2SO_3, a method modified slightly from that of Ripper is used. To the sample (25 ml) is added 10 ml of N-HCl and 1 ml of starch soln. (1%), and the soln. is titrated with 0·01N-iodine. For a precise determination of free SO_2, the polarographic procedure is recommended (apparatus illustrated), and results are quoted.
B. B. BAUMINGER

3480. **Manufacture of modern edible fats and a contribution to their analysis.** W. Wachs (Inst. für Lebensmittelchemie und Lebensmitteltechnol., Tech. Univ., Berlin-Charlottenburg). *Z. LebensmittUntersuch.*, 1960, **113** (3), 213–222.—The chemical and physical changes involved in the conversion of natural oils and fats into edible fats are reviewed. Procedures available for the analytical detection of these changes are discussed, and a brief outline is given of chromatographic methods for determining lauric acid in the examination of chocolate fats for small amounts of palm-kernel fats, for detecting palmitic acid esterified in a hardened fat, and for separating the mixture of mono- and di-carboxylic acids obtained on oxidation of mono-unsaturated acids in the detection of hydrogenated or elaidinised constituents. (37 references.) C. L. HINTON

3481. **Analysis of complex lipid mixtures by thin-layer chromatography and complementary methods.** D. C. Malins and H. K. Mangold (Bureau of Commercial Fisheries, U.S. Fish and Wildlife Service, Seattle, Wash.). *J. Amer. Oil Chem. Soc.*, 1960, **37** (11), 576–578.—Thin-layer adsorption chromatography on silicic acid has been applied to the routine separation of lipids into groups; further resolution into individual components was achieved by gas - liquid or reversed-phase partition chromatography on siliconised silicic acid plates (chromatoplates), prepared by slowly immersing thin-layer silicic acid plates in 5% silicone in ethyl ether and evaporating off the solvent. With menhaden oil fractions, spots were developed in 40 min. with methyl cyanide - acetic acid - water (14:2:5) and detected with α-cyclodextrin - iodine. Batyl alcohol (R_F 0·38) and selachyl alcohol (R_F 0·45) were separated from ratfish-liver oil fractions on siliconised paper developed for 18 hr. at 30° with tetrahydrofuran - water (3:2). P. M. KINGSTON

3482. **Determination of free fatty acids in fats and oils by non-aqueous titration method.** Toshiro Murata and Nagayuki Miyazaki (Pharm. Fac., Univ. of Kumamoto, Japan). *Chem. & Pharm. Bull., Japan*, 1961, **9** (1), 73–76 (in English).—A clear end-point, with the avoidance of cloudy emulsions, was obtained in this determination by a method described by Fritz and Lisicki (*Anal. Chem.*, 1951, **23**, 589). Metallic sodium (1·2 g) was dissolved, with cooling, in 16·7 ml of methanol, in the absence of CO_2, and 30 ml of methanol and 250 ml of benzene were added when dissolution was complete. This ≃0·1N-Na methoxide was standardised against benzoic acid, dissolved in 20 ml of a mixture (1:3) of methanol and benzene, in the absence of CO_2, with 0·1% thymol blue in methanol as indicator. For the determination of free fatty acids, a weighed amount of fat or oil was dissolved in 20 ml of the mixture (1:3) of methanol and benzene, and the soln. was titrated with standard Na methoxide soln. Results are recorded of determinations on samples of butter, arachis oil, rape oil and soya-bean oil. I. A. SIMPSON

3483. **Determination of the total oxidised acids in fatty materials.** M. Naudet, M. J. Perrot and P. Desnuelle [Lab. Nat. des Matières Grasses (I.T.E.R.G.), Marseille, France]. *Peint.-Pigm.-Vern.*, 1960, **36** (11), 634–637.—The combined fatty acids are subjected to reverse-phase partition chromatography with a column of high-mol.-wt. polyethylene, a liquid paraffin stationary phase and a mobile phase of aq. acetone. The acidity of the eluate is determined continuously by titration, and

oxidised acids, *e.g.*, keto- or hydroxy-fatty acids, are eluted in a distinct band. Results for the fatty acids of rape and grape-seed oil and of tallow are compared with those obtained on a granulated rubber column. L. A. O'NEILL

3484. **Determination of vitamin D in animal tissue.** V. N. Bukin and I. N. Garkina (Inst. Biochem., Acad. Sci., Moscow). *Biokhimiya*, 1961, **26** (1), 40–43.—*Procedure*—A chromatographic method is carried out on the sterol-free unsaponifiable matter. Vitamin A, carotene and other polyenes are separated from a light petroleum soln. on a column of alumina. The residue from the eluate is dissolved in methanol and subjected to ascending paper chromatography in light petroleum - acetone (9:1) together with three standards of pure vitamin D. The zones are located with u.v. light, extracted with $CHCl_3$ and treated with acetic anhydride and $SbCl_3$. The extinction is measured at 500 mμ against $CHCl_3$, and vitamin D is calculated from a standard curve. K. R. COOK

3485. **The influence of pectins on the shape of the polarographic wave of ascorbic acid.** Z. Bożyk and S. Krauze (Food Res. Dept., Acad. of Med., Warsaw). *Chem. Anal., Warsaw*, 1961, **6** (1), 75–82.—The adverse effects of pectins on the polarographic determination of ascorbic acid in deproteinised extracts from fruit and vegetables depend on their concn. and molecular size and are similar to those of typical surface-active agents. Specifically, the $E_{\frac{1}{2}}$ is shifted towards the positive and the wave height is lowered, the wave itself is flattened and the tangents, especially the limiting-current tangent, are steepened. Amounts of pectin >0·2% produced unmeasurable waves. It is concluded that deproteinisation of the extracts is often insufficient and pectin removal or dilution is necessary. For maximum accuracy, the method of standard additions is recommended, especially if a series of determinations is undertaken. P. BRYCH

See also Abstracts—**3147**, Modification of Luff-Schoorl copper reagent. **3191**, ^{90}Sr in milk. **3279**, Fe in potatoes. **3321**, Volatile carbonyl compounds in foods. **3345**, Pentachloronitrobenzene in cabbage. **3362**, Arsenic in malting coal. **3390**, Mg in foods. **3418**, Acetyl groups in pectin. **3422**, Ipomeamarone in sweet potatoes. **3453**, Differentiation of capsaicin from *N*-vanillylnonanamide.

Sanitation

Analysis of air, water, sewage, industrial wastes, industrial poisons.

3486. **Electrolytic determination of carbon monoxide [in air].** P. J. Ovenden (Nat. Coal Board, Glasgow Road, Edinburgh, Scotland). *J. Electroanal. Chem.*, 1961, **2** (1), 80–87.—Oxidation of CO at a vibrating micro-electrode (24 s.w.g. steel wire plated with palladium over a flashing of copper) gives, under specified conditions, a means for its determination. The cell used is an H-cell, one half of which contains palladium electrodes and through which H gas passes. The other half of the cell, through which the sample gas passes (10 ml per sec.), contains the vibrator. Both halves of the cell are filled with 0·1M-H_3BO_3, adjusted to pH 10 with NaOH, or 0·1M-H_3PO_4 adjusted to pH 7. The free wire length of the vibrating electrode is 14 cm, the frequency is 31 c/s, and the speed of movement 65 cm per sec. The electrode is intermittently polarised to the required potential by a special timing switch (described). The solubility of CO in phosphate and borate buffers, respectively, was 0·028 and 0·025 mg per ml. The diffusion coeff. were 2×10^{-5} and 5×10^{-5} sq. cm per sec., and diffusion currents were proportional to the concn. of CO up to this value. The method may be applied to continuous analysis. G. RUSSELL

3487. **Colorimetric method for continuous recording analysis of atmospheric fluoride. Test chamber and interference studies with the Mini-Adak analyser.** D. F. Adams, R. K. Koppe and N. E. Matzek (Div. of Ind. Res., State Univ., Pullman, Wash., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 117–119.—The suitability of a miniature Adak fluoride analyser, with Zr - Eriochrome cyanine R reagent, for the continuous analysis of the atmosphere in studies of fluoride fumigation and plant behaviour is investigated. The apparatus has an analysis efficiency of ≃18% for submicron-cryolite fumes. In the determination of 5 μg of F^- per cu. metre, SO_2 (<0·8 p.p.m.), H_2S (<0·9 p.p.m.), methanethiol (<0·8 p.p.m.), NO (<1·5 p.p.m.), NO_2 (<0·23 p.p.m.), O_3 (<1·7 p.p.m.), Al^{3+} (<50 μg per cu. metre), Mg^{2+} (<1777 μg per cu. metre), Ca^{2+} (<6777 μg per cu. metre), Na^+ (<6777 μg per cu. metre), PO_4^{3-} (<13 μg per cu. metre), SO_4^{2-} (<67 μg per cu. metre), Cl^- (<6777 μg per cu. metre), NO_3^- (<6777 μg per cu. metre), and NO_2^- (<6777 μg per cu. metre) do not interfere. J. P. STERN

3488. **Application of gas chromatography to analysis of solvent vapours in industrial air.** B. Levadie and J. F. Harwood (Vermont Dept. of Health, Barre, U.S.A.). *Amer. Ind. Hyg. Ass. J.*, 1960, **21** (1), 20–24.—Air samples containing added solvents were bubbled through *m*-xylene, and 0·05 ml was chromatographed on a Beckman GC-2 column at a temp. of 125° and a flow rate of 94 ml per min. Recoveries of the components of a mixture of added chlorinated hydrocarbons ranged from 97 to 106%. CHEM. ABSTR.

3489. **Methods of testing water used in industry.** British Standards Institution (2 Park Street, London, W.1). B.S. 2690 : 1956, Addendum No. 1 (1960). 27 pp.—The addendum deals specifically with the analysis of high-purity water and includes methods for the determination of solids content, CO_2, dissolved silica, total Fe and dissolved O.

3490. **Apparatus for use in measuring the residual hardness of water.** Energeticky Ustav [Inventor: A. Anscherlik]. Brit. Pat. 861,518; date appl. 15.1.59.—In the determination of the hardness of water by a colorimetric method in which samples of water, to which a suitable dye has been added, are tested at intervals, the intensity of the colouring of the soln. is converted into an electric value by means of a photo-electric cell. All the necessary operations are brought about automatically, and the apparatus comprises a switch for the timed distribution of electric impulses constantly applied to actuating relays for the accurate dosage of the water and the chemical reagents, and a recording millivoltmeter for indicating and recording the electric value. J. M. JACOBS

3491. **Suggested amperometric titration method for determining dissolved oxygen.** E. Goldman and B. Dietz (Dept. Water and Power, Los Angeles, Calif.). *J. Amer. Wat. Wks Ass.*, 1960, **52** (12), 1575–1576.—The end-point is more precise in the

method described than in the Winkler method. The cell of the titration apparatus is soldered in place, thus improving the connection and eliminating drift. *Procedure*—To the sample (300 ml) add 48% $MnSO_4.4H_2O$ soln. (2 ml) and aq. alkaline iodide soln. (70% of KOH and 15% of KI) (2 ml). Mix, and re-dissolve the ppt. by adding 1 to 2 ml of conc. H_2SO_4. To an aliquot (10 ml) add H_2O (190 ml) and acetate buffer soln. (pH 4) (1 ml) and titrate with 0·00564N-arsenosobenzene (prep. described). The standard deviation averaged ±0·1 over the range 1·5 to 8·0 p.p.m. of O. R. E. E.

3492. **Micro-determination of dissolved oxygen in water according to a modified Winkler method.** B. K. Lo (Lab. Elektr. Centr., Nieuwe Hemweg, Amsterdam, Netherlands). *Chem. Weekbl.*, 1960, **56** (52), 732–733.—The influence of the exposure to air of the soln. to be titrated is investigated. It is found that oxidation of KI by O from the air leads to erroneous results, especially when low O concn. are to be determined. It is therefore recommended that the titration be carried out with exclusion of air. M. J. Maurice

3493. **Gas-chromatographic determination of trace amounts of the lower fatty acids in water.** E. M. Emery and W. E. Koerner (Monsanto Chemical Co., St. Louis, Mo., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 146–147.—A flame ionisation detector is used in conjunction with a column packed with Tween 80 on Chromosorb W. The detector is insensitive to the water present, and sharp peaks are obtained for the fatty acids. G. P. Cook

3494. **Methods for the determination of small amounts of anionic surface-active materials in water effluents.** W. K. Fischer (Henkel & Cie G.m.b.H., Düsseldorf, Germany). *Fette, Seif., Anstrichmitt.*, 1961, **63** (1), 14–23.—Methods available for the determination of small amounts of anionic surface-active agents in water and effluents are reviewed and critically discussed. Some results obtained during the long experience of the authors are presented, and these show that the Longwell and Maniece method (*Analyst*, 1955, **80**, 167) is the most suitable for adoption as standard, and gives results of satisfactory accuracy for routine analysis. (46 references.) S. M. Marsh

3495. **Rapid analytical methods for the investigation of metals and inorganic raw materials. XII. Determination of traces of beryllium in mineral waters and mineralogical materials.** Z. Šulcek, J. Doležal and J. Michal (Geol. Inst. Prague, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1961, **26** (1), 246–254 (in German).—Traces of Be are quant. adsorbed on a column of silica gel over the pH range 4·5 to 9·5. The Be may be eluted with HCl and determined fluorimetrically with morin. In the presence of excess of F^- and PO_4^{3-} adsorption is complete at pH 6·5 to 9·5. Interference by most other elements can be prevented by means of EDTA or tartrate, but Sn, U, Sb, Th, Zr and Ti are adsorbed on the column and, if present in large excess, must be separated by modifications of the procedure. *Procedures*: (*i*) *For mineral waters*—Acidify the sample (1 to 10 μg of Be) with HCl, oxidise with 30% H_2O_2 (1 ml) and evaporate to dryness. Re-dissolve the residue in HCl (1 to 3 ml), dilute to 100 ml and filter. Neutralise the filtrate with aq. NH_3. After adding 0·2M-EDTA (30 ml), adjust the pH to 6·5 and pass the soln. through a silica-gel column (8 cm × 1 sq. cm) at a rate of 3 ml per min. After washing the column with H_2O (3 × 25 ml followed by 50 ml), elute the Be with 5 ml of HCl (1 : 1). Add a further 5 ml of acid, set aside in the column for 5 min., and wash the column with 50 ml of H_2O. Neutralise the combined soln. with 25% NaOH soln., and add 2·5N-NaOH (4 ml) and morin soln. (0·02% in acetone) (2 ml). Dilute to 100 ml and measure the fluorescence. (*ii*) *For mineralogically separated zinnwaldite* [*lithium biotite*]—Decompose the finely powdered sample (0·1 to 1 g) with Na_2CO_3 or $KHSO_4$, remove silica in the usual way, and make the soln. up to 250 ml. To an aliquot (1 to 10 μg of Be) add 0·2M-EDTA (20 to 30 ml), adjust the pH to 6·5, and proceed as described above. J. W. Price

3496. **Determination of uranium in sea water by pulse polarography.** G. W. C. Milner, J. D. Wilson, G. A. Barnett and A. A. Smales (Chem. Div., A.E.R.E., Harwell, England). *J. Electroanal. Chem.*, 1961, **2** (1), 25–38.—In the complex extraction procedure described, ^{237}U is used as a radioactive tracer to measure the chemical yield (by γ-counting) before recording the polarogram. For the polarography, a portion of the final extract in $HClO_4$, with Na tartrate as supporting electrolyte, is de-oxygenated for 15 min. with N purified with $CrSO_4$ soln. The U peak, *vs.* the mercury-pool anode, is at −0·39 V. The concn. of U is determined by the method of standard addition (4 μg of U in 0·25 ml of the same soln. as in the cell). The main error is due to the sloping and curved base line. The radiochemical yield varied from 75·3 to 91·2%. G. Russell

---

See also Abstracts—**3191**, ^{90}Sr in water. **3279**, Fe in water. **3317**, Aliphatic aldehydes in air. **3327**, Chloropicrin in air. **3364**, Detergents in sewage. **3540**, Electrostatic precipitator for aerosols.

Agricultural analysis

Soil, fertilisers, herbicides, pesticides, animal feeding-stuffs.

3497. **Detection of ions in vegetable matter by means of ion-exchange resins.** I. K. Tsitovich. *Izv. Vyssh. Ucheb. Zavedenii. Khim. i Khim. Tekhnol.*, 1959, **2** (6), 846–851; *Ref. Zhur., Khim., Biol. Khim.*, 1960, (20), Abstr. No. 28,086.—Concentration with cation-exchange resins KU-1 or SBS and anion-exchange resin AN-1 raises the sensitivity of the micro-crystalloscopic reaction for ions many times. A method is described for the detection of Cu^{2+} as $[Cu(C_6H_5NH_2).5H_2O](SCN)_2$, $[C_6H_2(NO_2)_3O]_2.[Cu(NH_3)_4]$ or $Cu[Hg(SCN)_4].H_2O$; Hg^{2+} as $[Hg(C_5H_5N)_4]Cr_2O_7$ or $Co[Hg(SCN)_4]$; Ba^{2+} as $BaWO_4$ or $BaCrO_4$; Zn^{2+} as $[Zn(C_5H_5N)_2](SCN)_2$, $Zn[Hg(SCN)_4]$ or $3Na_2CO_3.8ZnCO_3.8H_2O$; AsO_4^{3-} as $NH_4MgAsO_4.6H_2O$ or Ag_3AsO_4; and AsO_3^{3-} as Ag_3AsO_3. After adsorption of the ions the cation-exchange resins are treated by the appropriate reagents and examined microscopically. It is recommended that the colour reactions of the anions should be carried out directly on a micro-column. The method is used to detect insecticide ions in vegetable matter. K. R. Cook

3498. **Determination of lactic acid in diffusion juice.** A. Šepitka and E. Zradulová (Biochem. Dept., Chem. Inst., Acad. Sci., Bratislava, Czechoslovakia). *Listy Cukr.*, 1961, **77** (1), 16–20.—All acids present in the sample, including lactic acid, are isolated and separated from interfering substances

(proteins, carbohydrates) on a column of anion-exchange Wofatit L-150 (2 g for 25 ml of the sample containing $\gtrsim$100 mg of lactic acid). The acids are eluted with aq. NH_3, which is removed by distillation. Lactic acid is oxidised with $KMnO_4$ and the acetaldehyde formed is distilled into a soln. of $NaHSO_3$, the excess of which is determined iodimetrically. The results are 1% below theoretical values, but this systematic error can be eliminated by using a nomogram for the evaluation.

J. ZÝKA

3499. **Determination of sugar and starch content of vines by means of the anthrone reagent.** M. Pánczél and J. Eifert (Lab. f. Rebenforschung, Staatsgut Balatonboglár, Hungary). *Mitt. Wein- u. Obstbau, Wien, A*, 1960, **10** (3–4), 102–110.—The determinations serve to assess the nutrient content of year-old shoots used for propagation. A sample (0·5 g) of the dried finely ground material is extracted with hot 80% ethanol (4 × 5 ml), the first extraction being made by grinding with sand (0·5 g); the combined centrifuged extracts are treated first with neutral Pb acetate soln. to remove polyhydric phenols, and then with Na_2HPO_4 soln. to remove the excess of Pb; the sugars are determined colorimetrically in the (filtered and diluted) soln. by Clegg's anthrone method. Starch is similarly determined in a centrifuged and diluted extract of the residue from the previous extraction made with 52% $HClO_4$ soln. (2 × 6·5 ml). The colorimetric comparisons are made with standard soln. of glucose. Determinations by these methods are more accurate and rapid than those by the Bertrand, Hagedorn-Jensen, Popoff and Perlusz-Molnár methods. The accuracy is within $\pm$0·18% for sugar, and $\pm$0·17% for starch.

P. S. ARUP

3500. **Quantitative determination of zinc in soils by a spectrographic method.** E. A. Pometun and V. I. Boyarova (Inst. of Chem., Acad. Sci., Tadzhik SSR, Stalinabad). *Zhur. Anal. Khim.*, 1961, **16** (1), 103–105.—Zinc is distilled from the crater of a small special electrode containing 200 mg of soil and 200 mg of a mixture of carbon with 10% of CdI_2. By using the line pairs Zn I 3345·02 and Cd I 3133·17, or Zn I 3282·33 and Cd I 3133·17, it is possible to determine 0·002 to 0·01% of Zn in soil with an error $\gtrsim\pm$8%.

A. BURWOOD-SMITH

3501. **Rapid method for the determination of potassium in industrial fertilisers.** V. Novák and M. Mátl (Dept. Anal. Chem., Inst. Chem. Tech., Pardubice, Czechoslovakia). *Sborn. Věd. Prací, Vysoké Školy Chem. Technol., Pardubice*, 1960, 129–133.—*Procedure*—Heat the sample (2 g) with H_2O (50 ml) and conc. HCl (10 drops) for 5 min. Add H_2O (80 ml) and boil for 30 min. Filter the hot soln., wash the residue with H_2O and dilute the filtrate to 250 ml. Pass 25 ml through a column of anion-exchange resin, Anex L (OH^- form) (1 ml per min.), wash the resin with H_2O (75 ml) until the washings are free from alkali, and titrate the eluate and washings with 0·1N-HCl, with methyl orange as indicator. Evaporate the liquid to dryness in a platinum dish, ignite the residue at 450°, dissolve it in H_2O (2 ml), transfer with H_2O (10 ml) to a 50-ml flask and add an amount of Ag_2O approx. equivalent to the amount of alkali metals, calculated from the titration. Shake for 1 min., add ethanol (30 ml) and shake for 1 min. Filter through a sintered glass filter G3 and titrate conductimetrically with $HClO_4$ as previously described (Novák and Krajina, *Anal. Abstr.*, 1959, **6**, 2032).

J. ZÝKA

See also Abstracts—**3179**, Be in soil. **3184**, Ca in plant and soil extracts. **3187**, Ca in fertilisers. **3233**, P_2O_5 in superphosphate. **3345**, Pentachloronitrobenzene in cabbage. **3364**, Alkylbenzenesulphonates in sewage. **3419**, **3420**, Gibberellic acid in plants. **3423**, Lime-sulphur deposits on plants.

5.—GENERAL TECHNIQUE AND APPARATUS

General

3502. **Thermistor micro-method for molecular weight.** A. Wilson, L. Bini and R. Hofstader (American Cyanamid Co., Bound Brook, N.J., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 135–137.—A simple apparatus for routine determinations, which is essentially a twin thermistor bridge capable of detecting a temperature difference of $1·5 \times 10^{-4}$°, is described. Both internal calibration and standard calibration techniques have been employed. The amount of sample of molecular weight 200 that would be required varies from 13·6 mg in H_2O (the least sensitive solvent) to 1·37 mg in CCl_4 (the most sensitive solvent), per determination.

E. G. CUMMINS

3503. **One-mark pipettes.** British Standards Institution (2 Park Street, London, W.1). B.S. 1583:1961. 12 pp.—This standard covers pipettes for 0·5 to 200 ml. The capacity is based on allowing no drainage time.

3504. **A simple and inexpensive technique for ultrafiltration of large and small quantities.** F. Rønnike (Royal Vet. and Agric. Coll., Copenhagen, Denmark). *Scand. J. Clin. Lab. Invest.*, 1959, **11** (4), 402–404.—The construction of an apparatus is fully described in which dialysis tubing (Visking), protected by gauze to withstand the maximum pressure produced by a water pump ($\simeq$25 torr) is used. This apparatus has a filtering surface of 200 sq. cm, and a filtering velocity of 150 to 200 ml per 24 hr.

D. B. PALMER

3505. **Improvements in or relating to processes and apparatus for determining the presence of combustible substances in a gas.** O. H. Drager. Brit. Pat. 861,467; date appl. 24.6.57. Germany, date appl. 23.6.56.—The amount of combustible substance, *e.g.*, methane, in a sample of gas is determined by causing the sample to undergo a compression-ignition phase in a chamber (at $\simeq$100 atm. and a compression ratio of $\lesssim$80:1) and then analysing the combustion, *e.g.*, by determining the quantity of heat produced during the combustion or the content of combustion products in the outgoing gas.

J. M. JACOBS

3506. **An automatic melting-point recorder.** L. F. Berhenke (Edgar C. Britton Res. Lab., The Dow Chemical Co., Midland, Mich., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 65–67.—Measurement of temperature is by means of a thermocouple which acts as a piston, resting on the solid in a standard capillary, 2 mm in diameter. Movements of this piston during the course of melting are traced on a drum and the corresponding temperatures are recorded. The temperature accuracy of the method is essentially that of the recorder (for the Honeywell

Brown ElectroniK used here, ±0·5°). The interpretation of the results obtained is discussed.
E. G. CUMMINS

3507. **Impurity determination by thermal analysis. I. The melting curve of a gradually frozen sample.** H. F. van Wijk and W. M. Smit (Inst. Phys. Chem. T.N.O., Utrecht, Holland). *Anal. Chim. Acta*, 1960, **23** (6), 545–551.—Based on the assumption of local equilibrium, an equation is established for the relation between temp., heat content and liquid fraction (y) in a binary solid - liquid system which is slowly frozen (or melted), and its application to the determination of impurities (*i.e.*, of y) by utilising the heating (melting) curve is described. The validity of the equation was confirmed experimentally for solid soln. of biphenyl - phenanthrene (0·2 to 0·8%) and Sn - Bi (0·3%). An equation based on the assumption of total equilibrium is less accurate. The concn. of impurity is determined either by the absolute method (which requires only one curve and combines the data therefrom with thermodynamic constants) or by the comparative method (which requires a melting curve of the sample and also one of the sample containing a known concn. of the impurity). W. J. BAKER

Chromatography, ion exchange, electrophoresis

3508. **Porous glass chromatography.** H. L. MacDonell (Res. and Dev. Div., Corning Glass Works, Corning, New York, U.S.A.). *Nature*, 1961, **189**, 302–303.—Chromatographic separations of fountain-pen inks have been achieved with porous glass (pore diameter $\simeq 4$ mμ) as medium. Particular advantages of porous glass are the inertness, the high surface area which confines the sample to a small area, and the ease of making optical measurements on the chromatogram. A. R. ROGERS

3509. **Quantitative chromatographic methods. II. An approach to paper chromatography [of amino-acids and peptides] of improved resolving power and reproducibility.** C. S. Hanes (Dept. of Biochem., Univ. Toronto, Ontario, Canada). *Canad. J. Biochem. Physiol.*, 1961, **39** (1), 119–140.—The problems involved in developing an improved and more comprehensive system of analysis of amino-acids and peptides by paper chromatography are considered. A chromatographic unit (described and illustrated) is designed to provide closer control of the physical conditions affecting chromatography and is contained in a constant-temp. cabinet equipped with a vapour-exhaust system. The chromatogram consists of buffer-impregnated Whatman No. 3 paper with a feeder wick stitched to the top and an absorption pad of folded filter-paper clamped to the bottom edge. In the impregnation of the paper, 35 sheets are suspended in the buffer soln., which is allowed to ascend by capillary action for 16 to 20 hr. The sheets are then placed on a rectangular perforated platform and the excess of buffer is removed by slight suction before the papers are air-dried in a clean atmosphere. Full details are given for assembling and spotting the chromatograms.

III. Factors controlling the patterns of separation of the amino-acids on paper chromatograms. E. H. M. Wade, A. T. Matheson and C. S. Hanes. *Ibid.*, 1961, **39** (1), 141–161.—The separation of amino-acids on one-dimensional chromatograms is studied with systems of buffered miscible solvents and papers pretreated with the same buffers. The solvents tested included methanol, ethanol, n-propanol, isopropyl alcohol, t-butyl alcohol, acetone and ethyl methyl ketone, and the buffer soln. included oxalate, succinate, tartrate, citrate, maleate, carbonate, phosphate and pyrophosphate. Results are given for the systematic variation in propanol - ethanol - aq. pyrophosphate systems of (*i*) solvent composition, (*ii*) pH of the buffer soln., (*iii*) additions of NaCl to the solvent and paper and (*iv*) composition of the conditioning liquid. The best system found was n-propanol - ethanol - aq. pyrophosphate (2 : 1 : 1). The buffer (pH 8·9 ± 0·05) consisted of 22·31 g of $Na_4P_2O_7.10H_2O$, 6·5 ml of N-HCl and 5·44 g of NaCl, made up to 1 litre with water. This system separates 13 to 15 amino-acids from a mixture of 18; the pairs aspartic acid - cysteic acid and serine - glycine are not separated but can be resolved in a phenol - aq. borate system at pH 9·3.

IV. Stabilised chromatographic systems of high resolving power for amino-acids. C. S. Hanes, C. K. Harris, M. A. Moscarello and E. Tigane. *Ibid.*, 1961, **39** (1), 163–190. **Appendix: Note on moisture relations of chromatographic papers.** C. S. Hanes and D. D. Wood. *Ibid.*, 1961, **39** (1), 190–194.—Optimum conditions are given for the successful use of two complementary systems in one-dimensional chromatographic separations of amino-acids. The propanol - ethanol - pyrophosphate system is used with a propanol - tartrate system and buffer-impregnated papers. The chromatograms obtained with these systems under varied conditions are illustrated. In the preconditioning of the chromatographic units, consideration is given to the water content of the paper introduced into the units. The water content of buffered and unbuffered papers can be deduced from the humidity of the ambient atmosphere. Before the introduction of the solvent during the pre-conditioning period, each chromatographic unit with a given solvent is adjusted so as to contain the same total amount of water (added water plus water in the paper). This adjustment of water content is important for obtaining reproducible chromatograms. E. C. BUTTERWORTH

3510. **A sample-solvent evaporator for paper chromatography.** F. M. Ganis (Dept. of Radiation Biology, Univ. of Rochester Sch. of Med. and Dentistry, N.Y., U.S.A.). *J. Chromatography*, 1961, **5** (1), 84–85 (in English).—This simple apparatus blows air on to the lower surface of the paper at the same time that the sample is applied to the upper surface. A. R. ROGERS

3511. **The bar-oven, an aid for microchemistry. I. A simple method for concentrating the spots on chromatograms.** H. Reimers (Monheimerstr. 26, Köln-Weidenpesch, Germany). *Mikrochim. Acta*, 1961, (1), 140–144 (in German).—The material, on a wedge-shaped paper, is eluted in a heated zone, so that solvent is rapidly removed; a round spot is thus concentrated in a band at the narrow end of the paper. Best results are obtained when the temp. of the oven is 5° to 10° higher than the b.p. of the solvent. A concentration of the test substance of $\simeq 100$-fold can be achieved, with a corresponding increase in sensitivity. The process can be carried out under vacuum or in an inert gas. The apparatus is described.

II. A simple method for transferring a substance or a mixture from one chromatogram to the starting point of another. H. Reimers. *Ibid.*, 1961, (1), 145–148 (in German).—After concentration of the material as described in Part I, the band is cut out

and laid on the base line of a fresh piece of chromatographic paper, which is then placed in position over an 8-mm hole in the ring-oven. The material is transferred by extraction with micro drops of solvent, as for the ring-oven technique.

III. Qualitative analysis by extraction on the paper. H. Reimers. *Ibid.*, 1961, (1), 149–153 (in German).—The strip is dipped in a suitable solvent, which on ascending (or descending) the chromatogram is rapidly removed by heat from the oven. Advantages over the use of the ring-oven include a 10 to 50-fold enrichment of material during the first extraction, and the possible use of specific solvents for each component of the mixture. A scheme for the separation of a 4-component system is proposed.
P. D. PARR-RICHARD

3512. **Device for spraying paper chromatograms.** L. Reio (Wenner-Gren Inst., Dept. of Metabolic Res., Univ. of Stockholm, Sweden). *J. Chromatography*, 1961, **5** (1), 86–88 (in English).—The cabinet described previously (*cf. Anal. Abstr.*, 1959, **6**, 1863) has been modified. Twenty series of 6 chromatograms each can now be sprayed consecutively.
A. R. ROGERS

3513. **The speed of analysis by gas chromatography.** J. H. Knox (The Univ., West Mains Road, Edinburgh, Scotland). *J. Chem. Soc.*, 1961, 433–441.—The minimum analysis time for packed or capillary columns for gas chromatography is discussed in terms of five independent variables—the column length, the column radius (for a capillary) or the particle radius (for a packed column), the thickness of the liquid film (or the ratio of film thickness to radius), the linear gas velocity, and the solubility of the substance being chromatographed. It is shown that the minimum time for elution is comparable in the two cases for a given separation factor, and choice of method can be made on other considerations. For the highest speed, the gas velocity to give the minimum HETP must be used. Hydrogen and He give faster analysis than N or Ar provided that the more severe solubility requirements can be met. Both methods require very thin coatings for maximum speed, and it is considered that non-porous packings such as glass beads or nylon are preferable to firebrick.
E. J. H. BIRCH

3514. **Adsorption phenomena and their effects on analytical accuracy in gas chromatography.** A. Weinstein (Dept. of Fuel Technol., The Pennsylvania State Univ., University Park, U.S.A.). *Anal. Chem.*, 1961, **33** (1), 18–22.—The theoretical and empirical implications of errors that can be shown to apply to peaks of short retention time are discussed.
E. G. CUMMINS

3515. **Gas chromatography: the effect of gaseous diffusion on mass transfer in packed columns.** R. Kieselbach (Engng Res. Lab., E.I. du Pont de Nemours & Co., Wilmington, Del., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 23–28.—Modification of the van Deemter equation, a three-term equation, which relates the height of an equivalent theoretical plate at a point along the length of a column to the carrier-gas velocity at that point, so as to embrace two further gas-diffusivity terms is discussed. The importance of the effect of gaseous diffusion on the theory of columns other than the capillary type, for which it is already accepted, is emphasised, and the need for a more rigorous theoretical treatment is stressed.
E. G. CUMMINS

3516. **Water-cooled sample-injection port for high-temperature gas chromatography.** P. G. Elsey and T. Rye (Ethyl Corp., Res. Lab., Detroit, Mich., U.S.A.). *J. Chromatography*, 1961, **5** (1), 88–89 (in English).—The rubber septum through which the sample is injected is brought outside the flash heater by means of a water-cooled spacer. A flow of 2 or 3 ml of water per min. keeps the septum at room temp. when the flash heater is at 350°. There is no effect on retention times or peak shapes.
A. R. ROGERS

3517. **A compact, easily cleaned and packed column for gas - liquid partition chromatography instruments.** J. R. Carew (Chem. Phys. Res. Lab., The Dow Chemical Co., Midland, Mich., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 156–157.—The performance of a standard 10-ft. column of $\frac{1}{4}$-in. stainless-steel tubing has been reproduced by the grooved-plate column described.
E. G. CUMMINS

3518. **Applications of gas - liquid chromatography. Collection of fractions from the gas chromatograph and their identification by infra-red spectroscopy.** J. Haslam, A. R. Jeffs and H. A. Willis (I.C.I. Ltd., Plastics Div., Welwyn Garden City, Herts., England). *Analyst*, 1961, **86**, 44–53.—Methods that have proved useful in the separation of milligram amounts of material by gas - liquid chromatography and i.r. identification of the separated products are described, with details of some of the apparatus used. Examples are given of the use of the method for investigating analytical problems arising in the plastics industry.
A. O. JONES

3519. **An enrichment trap for use with capillary columns.** R. Self (Res. Estab., Min. of Agric., Fish. and Food, Aberdeen, Scotland). *Nature*, 1961, **189**, 223.—The U-shaped trap described consists of 10 in. of nylon capillary, 0·01 in. in diam., coated with 15% tritolyl phosphate. Volatile substances obtained when potatoes are boiled in water are passed through the trap, immersed in liquid oxygen, after removal of the water vapour. The trap is then connected to a 50-ft. nylon capillary, diam. 0·01 in., coated with 15% tritolyl phosphate, and brought to room temperature, and the contents are then blown on to the column. Good separation of the components is obtained.
C. B. BAINES

3520. **Experiments with capillary column - flame ionisation detectors in gas chromatography.** I. Halász and G. Schreyer (Inst. Phys. Chem., Univ., Frankfurt-am-Main). *Chem.-Ing.-Tech.*, 1960, **32** (10), 675–685.—Full descriptions are given of a gas-chromatography (micro) method and apparatus in which a long (>100 metres) capillary tube (diam. ≃0·3 mm) is used for effecting the separation of an organic mixture (by partition between a liquid phase and the mobile gaseous mixture of sample and carrier gas), instead of a filled column, and in which detection and determination of the separated components are effected by arc recording flame-ionising detector, in which the flame is produced by combustion of dry H and component vapour in dry air. The capillary column gives more efficient, far reaching, and rapid separations than a filled column, but it requires a far higher (1000-fold) sensitivity of detection for the separated components, hence the use of the flame-ionising method. Operating details are fully discussed, including suitable liquids for the liquid phase (applied by wetting the capillary), and suitable ratios of substance vapour to carrier gas and of

vapour mix to liquid phase. A description is given of the separation of individual hydrocarbons from a light petroleum of boiling-range 100° to 140°, individual hydrocarbons from a mixture of 25 of them, and individual isomers from a mixture of *o*-, *m*- and *p*-xylenes (a separation not obtained with a filled column). H. L. WHITEHEAD

3521. **Recorder - integrator errors in gas-chromatography-area measurements.** C. H. Orr (Miami Valley Lab., The Procter and Gamble Co., Cincinnati, Ohio, U.S.A.). *Anal. Chem.*, 1961, **33** (1), 158–159.—Correction for errors due to non-linearity of both the recorder and the integrator is discussed. The tabulated correlation of peak height and error in area reported for the Wheelco Model 800-4600-S266 recorder when coupled to the Perkin Elmer Model 194 printing integrator was obtained by comparing the area taken out of the recorder - integrator combination to that put in. Once the relationship between millivolts of input to recorder deflection is known, the constants of the equations given can be determined, and such a table can be drawn up for any recorder - integrator combination. E. G. CUMMINS

3522. **Paper electrophoresis: temperature, paper wetness and serum-component mobility with the Kunkel system.** N. F. Hollinger and R. K. Lansing (School of Public Health, Univ. of California, Berkeley, U.S.A.). *J. Chromatography*, 1961, **5** (1), 38–45 (in English).—The variability of mobility between runs shows a relative standard deviation of $\simeq$6·5% for albumin and $\simeq$19% for γ-globulin. The observed mobility increases with increasing temp. and wetness; the latter effect is due to increase in electrolyte flow, and when corrected by a dextran marker the mobility values are independent of wetness. A. R. ROGERS

3523. **Simple techniques for starch-gel electrophoresis.** A. W. B. Cunningham and O. Magnusson (Tissue Dynamics Lab., Path. Dept., Univ. of Texas Med. Branch, Galveston, U.S.A.). *J. Chromatography*, 1961, **5** (1), 90–91 (in English).—Simple apparatus and techniques are described which provide forms for the gel and which prevent evaporation from and undue temp. changes in the gel during electrophoresis. A. R. ROGERS

3524. **Contact prints of starch-gel electrophoresis patterns.** E. W. Johns (Chester Beatty Res. Inst., Royal Cancer Hospital, London). *J. Chromatography*, 1961, **5** (1), 91–92 (in English).—Soak the stained gel in ethanol and then in a mixture of benzyl alcohol and glycerol (2:1) and place the stained surface in a pool of the mixture on extra-hard contact paper. Pour more mixture on to the back of the gel and apply a piece of plate glass. Expose and then develop with a contrast developer. A. R. ROGERS

See also Abstracts—**3301**, Identification of org. compounds by gas chromatography. **3332**, Thin-film electrophoresis and chromatography.

Optical

3525. **Improvements in and relating to grating monochromators.** Sir Howard Grubb Parsons & Co. Ltd. [Inventor: A. E. Martin]. Brit. Pat. 861,259; date appl. 5.8.58.—A grating dispersive element for a monochromator comprises a number of plane diffracting areas in parallel planes, each area being ruled with equi-spaced grooves, the groove spacings for the separate areas bearing a simple numerical relationship to one another (1:1, 1:2, etc.) with all grooves parallel and a different groove angle being associated with each area. J. M. JACOBS

3526. **Improvements in and relating to grating spectrometers.** Sir Howard Grubb Parsons & Co. Ltd. [Inventor: A. E. Martin]. Brit. Pat. 861,639; date appl. 19.3.57.—An infra-red monochromator is provided in which one or more diffraction gratings of the reflecting type, and having a saw-tooth groove form, are used as radiation dispersion means. One of the gratings is used simultaneously as wavelength-discrimination means for retaining the first order and suppressing the second and higher orders in respect of a selected wavelength. The arrangement is such that the diffracted radiation from this grating lies on the same side of a normal to the surfaces of an individual groove as the incident radiation, and the smaller of the two angles made by the incident and diffracted radiation with the normal has a value in degrees $>\lambda N/5000$, where λ is the first-order wavelength to which the grating is set and N is the number of lines per in. on the grating. J. M. JACOBS

3527. **Spectral analysis with a sifter electrode. II. The influence of alkali-metal halides on excitation.** J. Czakow and J. Minczewski (Inst. of Nucl. Res., Warsaw, Poland). *Chem. Anal., Warsaw*, 1960, **5** (6), 863–874.—A study of the vanadium spectrum shows that—(*i*) The effect of the concn. of extraneous metals is relatively insignificant but inclusion of an appropriate internal standard considerably improves the results. (*ii*) The effect of halogens is small enough to be practically eliminated by the use of an internal standard. (*iii*) Alkali-metal halides, in amounts about equal to that of the test element, increase the intensities of both spark and arc lines, thus increasing detectability. (*iv*) A large excess (1000-fold) of alkali-metal halides behaves as a spectrographic buffer. (*v*) The discharge acquires increasingly arc-like characteristics with increase in the atomic number of the alkali metal. (*vi*) The choice of the internal standard should be based on both line suitability and physical properties, which should be similar to those of the substance to be determined. The extrapolation method (standard additions) should be used with non-homogeneous samples (ores and minerals), as it reduces the effect of impurities and simplifies the choice of internal standard by permitting utilisation of any neighbouring lines. P. BRYCH

3528. **New spectrographic method for measuring variable spectral-line intensities in one [discharge] cycle.** J. Roliński (Central Bureau of Measures, Warsaw, Poland). *Chem. Anal., Warsaw*, 1960, **5** (6), 951–965.—A narrow beam of radiation from a mercury lamp (S500) was time-selected with respect to the cycle by a millicycloscope (a synchronised rotating shutter) and recorded spectrographically. Spectrograms were taken at 30° intervals and radiation intensities at 2537 A, 2967 A, 3663 A and 4339 A were analysed in conjunction with current and potential variations during a cycle, obtained on an oscillograph. Significant differences in current - voltage asymmetries were noted and these undoubtedly affected similar differences in optical asymmetries, which also differed between themselves. P. BRYCH

3529. **A simple anaerobic cell for the Beckman spectrophotometer.** B. J. McCormick and G. Gorin (Dept. of Chem., Oklahoma State Univ., Stillwater, U.S.A.). *Anal. Chem.*, 1961, **33** (1), 157.—The modification of a standard rubber vaccine-bottle stopper (Sargent S-9075) to fit standard spectrophotometer cells and cell-holders and to permit flushing with an inert gas and filling by hypodermic syringe is described. E. G. CUMMINS

3530. **A spectrometer for the far infra-red.** R. H. Wright and P. N. Daykin (British Columbia Res. Council, Vancouver 8, B.C., Canada). *Nature*, 1961, **189**, 212.—A simple spectrometer, easy to construct and with good light-gathering power, is described. It is based on a Fresnel zone plate as the focusing and dispersing element with a chopped-light source, giving first-order spectra, and a bolometer detector moving on a straight track to scan the spectrum. The system is free from spherical aberration at 68 μ, and the zone plate has a focal length of 73·5 cm at this wavelength. There are no collimating mirrors or movable slits and the spectrometer is constructed for use between 35 and 80 μ. C. B. BAINES

3531. **Simple adjustable cell for infra-red spectroscopy.** J. Suchý (Dept. Nat. Products, Chem. Inst., Acad. Sci., Bratislava, Czechoslovakia). *Chem. Zvesti*, 1961, **15** (1), 70–73.—The cell described is adjustable to an accuracy of 0·001 mm. J. ZÝKA

3532. **The use of indene for the calibration of small infra-red spectrometers.** R. N. Jones, N. B. W. Jonathan, M. A. MacKenzie and A. Nadeau (Div. of Pure Chem., Nat. Res. Council of Canada, Ottawa). *Spectrochim. Acta*, 1961, **17** (1), 77–81.—Accurate measurement of the position of 77 bands in the spectrum of indene in the range 4000 to 650 cm^{-1} is reported. The bands have been measured on a grating instrument with a precision of $\pm$3·0 to $\pm$0·2 cm^{-1}. E. G. CUMMINS

3533. **Scavenging system for the sample cell of an infra-red gas analyser.** J. F. Kemp (Nat. Mech. Engng Res. Inst., S. African C.S.I.R., Pretoria). *Anal. Chem.*, 1961, **33** (1), 159–160.—Contamination due to the previous sample has been reduced to 5×10^{-4} % by one operation of the cycle described without the use of vacuum or large amounts of sample. E. G. CUMMINS

3534. **Micro-technique for the infra-red study of solids. Diamonds and sapphires as cell materials.** E. R. Lippincott, F. E. Welsh and C. E. Weir (Dept. of Chem., Univ. of Maryland, College Park, U.S.A.). *Anal. Chem.*, 1961, **33** (1), 137–143.—A cell designed for use with a beam-condensing unit is described. Diamond, which always absorbs between 2700 and 1800 cm^{-1}, and sapphire, which absorbs at $<$1600 cm^{-1}, have been used as cell materials. Most solids can be examined at the $<$1·0-mg level simply by increasing the pressure to which they are subjected until they are transparent. A nominal pressure of several thousand atmospheres may be achieved between diamond windows by simple adjustment of the pressure plate of the cell. E. G. CUMMINS

3535. **Applications of complementary tri-stimulus colorimetry. II. A least-squares method applied to multicomponent systems.** H. Flaschka (School of Chem., Georgia Inst. of Technol., Atlanta, U.S.A.). *Talanta*, 1961, **8** (1), 8–15.—It is shown that the calculation of colorant concn. in complementary tri-stimulus colorimetry can be improved by the application of the method of least squares rather than by simple averaging of sets of results. In this improvement a matrix approach is especially useful and the required equations are developed. Comparison of results obtained by the two methods shows the superiority of the matrix approach. C. B. BAINES

3536. **Micro-photometric titration of substances in the ultra-violet region with end-point recording. I. Construction of a micro-titrimeter and the determination of iron content.** K. P. Stolyarov, N. N. Grigor'ev and Kuo-Liang Ch'ên (A. A. Zhdanov Leningrad State Univ.). *Zhur. Anal. Khim.*, 1961, **16** (1), 4–7.—The design of a micro-titrimeter for micro-photometric titrations in the u.v. region is suggested. The determination of Fe in bronze, manganese concentrates and ore is taken as an example with a relative error of $\simeq$0·5%. The iodimetric determination of Fe is based on the reaction—

$$2Fe^{3+} + 2I^- \rightarrow 2Fe^{2+} + I_2,$$

the iodine formed being back-titrated with $Na_2S_2O_3$ soln. A. BURWOOD-SMITH

3537. **The Gorbach - Lange micro-capillary photometer.** G. Gorbach and C. Weber (Inst. f. biochem. Technol., Lebensmitt. u. Mikrochem., Tech. Hochschule, Graz, Austria). *Fette, Seif., Anstrichmitt.*, 1961, **63** (1), 23–25.—Details are given of an improved micro-capillary photometer having a 100-mm capillary tube and capable of the quant. determination of μg amounts of substances with an accuracy of $\pm$1%. S. M. MARSH

3538. **The fluorescence determination of uranium. I. Design of equipment and testing.** R. Fernandez Cellini, F. de la Cruz Castillo and R. Barrera Pinero (Junta de Energia Nuclear, Madrid, Spain). *U.S. Atomic Energy Comm.*, Rep. JEN-67-DQ/1-21, 1960. 20 pp.—Equipment for analysis by fluorescence was developed in order to determine U at a lower concn. than could previously be determined by the most sensitive analytical method. A fluorimeter was adapted to measure the fluorescence emitted by a bead of $NaF - Na_2CO_3 - K_2CO_3$ excited by u.v. light of 365 mμ. The intensity of the light emitted was measured with a RCA 5819 photomultiplier and associated electronic equipment. The fluorimeter gives a linear response for amounts of U between 0 and 15 μg with a sensitivity of 0·001 μg of U. NUCL. SCI. ABSTR.

3539. **New design of spectropolarimeter.** E. J. Gillham and R. J. King (Nat. Phys. Lab., Teddington, Middx.). *J. Sci. Instrum.*, 1961, **38** (1), 21–25.—Details are given of the design and performance of a spectropolarimeter in which the functions of monochromator and polarimeter are combined by using two crystalline quartz prisms to disperse the radiation and to polarise it. The prisms are in fixed positions and wavelength scanning is achieved by rotation of two plane mirrors about a common axis. The optical rotation of the specimen is compensated by a Faraday cell, and is thus measured in terms of current. A modulator between the polariser and analyser oscillates the plane of vibration of the polarised light through a suitable half-shadow angle at an audio-frequency. A departure from the balance position causes a modulation of the intensity of the emergent light

at the oscillation frequency; this is detected by a photomultiplier, the a.c. output of which is used to control the d.c. through the compensator. The instrument covers a wavelength range from 600 to 220 mμ. G. SKIRROW

See also Abstracts—**3142**, Applications of X-ray spectrography in industry. **3177**, Automatic X-ray spectrography. **3475**, Formazin standards for turbidity.

Electrical

3540. **A miniature electrostatic precipitator for sampling aerosols. Theory and operation.** M. Robinson (U.S. Army Chem. Corps, Fort Detrick, Frederick, Md., U.S.A.). *Anal. Chem.*, 1961, **33** (1), 109–113.—Equations are derived for describing the performance of the precipitator, and checked experimentally. J. P. STERN

3541. **Method and apparatus for detecting minute concentrations of gases and vapours.** Mine Safety Appliances Co. Brit. Pat. 860,576; date appl. 3.12.58. U.S.A., date appl. 5.12.57.—The gaseous mixture is passed through an ionisation chamber (*cf.* Brit. Pat. 860,577) and ionised in the presence of an electric field to produce an ionisation current. As the mixture enters the chamber it is exposed to a specific reagent that will react with the constituent to be detected to form nascent particles. The ionisation current in the chamber is measured before and after the mixture is exposed to the reagent. Hydrogen halides and org. acid vapours can be detected directly in parts per billion by using anhyd. NH_3 as the reagent and, conversely, NH_3, HCN and many amines can be detected with anhyd. HCl or acetic acid vapour as the reagent. Many halogenated hydrocarbons can be detected by pyrolysing the gas sample before it enters the chamber. J. M. JACOBS

3542. **Improvements in or relating to d.c. polarographs.** U.K.A.E.A. [Inventor: G. C. Barker]. Brit. Pat. 860,130; date appl. 26.6.57.—A residual current compensating circuit for a d.c. polarograph comprises means for applying a small a.c. voltage across the polarograph cell, means for deriving a d.c. voltage proportional to the a.c. current flowing in the cell, means for integrating the d.c. voltage, and means for combining a compensating current proportional to the integrated d.c. voltage with the d.c. current from the cell. The derived d.c. voltage at a fixed time in the life of each mercury drop is integrated, and a voltage proportional to the combined currents at this fixed time is fed to a recorder. J. M. JACOBS

3543. **Polarography in liquid ammonia above its critical temperature.** W. Hubicki and M. Dabkowska (Dept. of Inorg. Chem., Univ. Mariae Curie Skłodowska, Lublin, Poland). *Anal. Chem.*, 1961, **33** (1), 90–92.—The preparation and physical properties of the anhydrous liquid ammoniate of $LiClO_4$ are described. Polarography of soln. (10^{-3}M) of $PbSO_4$, $ZnCO_3$ and $CdCO_3$ over the temperature range 25° to 200° are reported. E. G. CUMMINS

3544. **Fused-salt polarography using a dropping-bismuth cathode.** R. J. Heus and J. J. Egan (Brookhaven National Lab., Upton, N.Y., U.S.A.). *J. Electrochem. Soc., New York*, 1960, **107**, 824–828.—An apparatus suitable for studying fused chlorides by the polarographic technique is described. Dropping bismuth is used in a way analogous to dropping mercury. Polarographic waves are shown for $PbCl_2$, $ZnCl_2$ and $CdCl_2$ in a LiCl - KCl eutectic at 450°. The method serves as an analytical tool at high temperatures and also yields physicochemical information such as diffusion coefficients by application of the Ilkovic equation. NUCL. SCI. ABSTR.

3545. **Determination of ultramicro concentrations of dissolved substances by the method of amalgam polarography on a stationary mercury drop.** A. G. Stromberg and E. A. Stromberg. *Zavod. Lab.*, 1961, **27** (1), 3–11.—A review with 51 references is presented. The discussion covers five types of mercury electrode, *viz*, a pendant drop obtained by transferring two or three mercury drops from a capillary to an amalgamated platinum wire in a glass tube, a sessile drop, a pendant drop obtained by forcing mercury out of a capillary tube by the turning of a screw, a drop obtained by electrolysis on to a platinum wire (0·1 mm × 0·1 mm) at 25 to 30 mA for 1 to 3 min., and mercury in a small glass cup at the end of an upturned capillary tube, together with studies of the effect of conditions of the preliminary cathodic deposition and those of the subsequent amalgam polarography on the depth of the anodic trough, the simultaneous determination of several elements, and methods of purifying and preserving reagents. G. S. SMITH

3546. **Device for increasing the sensitivity of a pH meter.** H. Jackson (Res. Lab., The British Aluminium Co. Ltd., Chalfont Park, Gerrards Cross, Bucks., England). *Analyst*, 1961, **86**, 76–78.—Certain titrations to a fixed-pH end-point show only a small increase in pH with increased addition of titrant at the end-point, *e.g.*, the titration of Al in sodium aluminate soln. when the OH^- released by addition of KF are titrated, and the titration of the boron - mannitol complex. The unit described, consisting of an auxiliary meter (100 μA) and a potential-divider circuit, is attached to a Pye universal pH meter or, with modification, to any pH meter having an external high-sensitivity socket. An eightfold increase in sensitivity is attained. A. O. JONES

3547. **Simple electrode system for measuring pH of blood.** G. L. Leathart (Med. Sch., King's Coll., Newcastle upon Tyne, England). *Lancet*, 1961, **i**, 88.—A glass-electrode assembly allowing blood to be introduced directly from the syringe is described. The apparatus can be placed in a water bath and is easily cleaned after use. The construction, from readily available materials, requires no special skill. H. F. W. KIRKPATRICK

3548. **Theory of chronopotentiometry with cylindrical electrodes. I. The quinone - quinol reaction at platinum and gold-wire electrodes.** D. G. Peters and J. J. Lingane (Dept. of Chemistry, Harvard Univ., Cambridge, Mass., U.S.A.). *J. Electroanal. Chem.*, 1961, **2** (1), 1–16.—An equation is derived for the transition-time constant for cylindrical wire electrodes, and its validity was tested and verified by using the results obtained for the reduction of quinone and the oxidation of quinol.

II. Application of theoretical transition-time relation in reduction of ferric iron and hydrogen ion. J. J. Lingane. *Ibid.*, 1961, **2** (1), 46–52.—These reductions were studied to verify further the equation derived in Part I. G. RUSSELL

3549. **Effect of electrode configuration and transition time in solid electrode chronopotentiometry.** A. J. Bard (Dept. of Chem., Univ. of Texas, Austin, U.S.A.). *Anal. Chem.*, 1961, **33** (1), 11–15.—Measurement of the transition-time constant $i_0\tau^{0.5}/C^\circ$, where i_0 is the current density (μA per sq. cm), τ is the transition time (seconds) and C° is the concn. of the electroactive species (millimoles per litre), with plane electrodes of different construction and orientation in the soln. over a very wide range of current densities, and with transition times ranging from 0·001 to 300 sec., is reported. It is concluded that $i_0\tau^{0.5}/C^\circ$ can be maintained constant to within $\pm 2\%$ by the use of a disc-type solid electrode sealed into glass tubing and mounted horizontally, with transition times ranging from 7 to 145 sec. E. G. CUMMINS

3550. **A liquid scintillation counter for continuous indirect measurement of beta activity in carbon dioxide and monoxide systems.** C. K. Hanson (Univ. Utah, Salt Lake City, U.S.A.). *Dissert. Abstr.*, 1960, **21** (4), 773.—Carbon dioxide is converted into CO by passage over molten zinc at 800°. The CO is converted into $COCl_2$, which is sol. in xylene, the solvent contained in the scintillation counter. The β-particles cause *p*-terphenyl dissolved in the xylene to fluoresce at 3400 to 3700 A. The light flashes are converted into voltage pulses by means of photomultiplier tubes. After proper amplification these are counted on a scaler. N. E.

3551. **Spectrometer for the determination of positron and gamma-ray cascade-emitting nuclides. [Radioactivation Analysis Symposium. Vienna, June 1959.]** K. Ljunggren (Royal Inst. of Technol., Stockholm 70, Sweden). *Pure and Appl. Chem.*, 1960, **1** (1), 31–34 (in English).—The scintillation spectrometer included two detectors from which the outputs were fed into a fast coincidence circuit with a resolving time of 28 mμsec.; the output from this circuit was fed into a triple coincidence circuit from which an output was obtained only if pulses were also present from the two detectors by way of slow pulse-height analysers. The ratio of the sensitivities for ^{60}Co:^{59}Fe was 52, for ^{64}Cu:^{137}Cs was $1{\cdot}2 \times 10^5$ and for ^{64}Cu:^{24}Na was 180. G. J. HUNTER

3552. **Neutron activation applied to geochemistry. [Radioactivation Analysis Symposium. Vienna, June 1959.]** W. Herr (Max-Planck-Inst. f. Chem., Mainz, Germany). *Pure and Appl. Chem.*, 1960, **1** (1), 35–52 (in English).—Work on the determination of the geological age of minerals is reviewed. The methods considered include the determination of ^{40}Ar after separation from materials containing K, the determination of ^{87}Sr in materials containing Rb, and the determination of ^{187}Os in materials containing Re. Neutron-activation methods are used, but a variant in the determination of the ^{187}Os is that the reduction in the specific activity induced in a mixture of natural Os and ^{187}Os is used as a measure of the ^{187}Os. The determination of U by neutron activation is briefly discussed, and the contents of Sc, He and Ne in iron meteorites are compared. G. J. HUNTER

3553. **Indirect neutron absorptiometry.** W. W. Meinke (Dept. of Chem., Univ. of Michigan, Ann Arbor, U.S.A.). *Talanta*, 1960, **5** (3–4), 264–266 (in English).—Elements such as Gd, Sm, Eu, Cd, Dy and B with high neutron-absorption cross-sections can be determined rapidly by neutron absorptiometry without destruction of the sample. It is also possible to determine small quantities of F^- and other ions that form insoluble compounds with Gd by measuring the neutron absorption of the pptd. compound. (*Cf. Anal. Abstr.*, 1961, **8**, 2726.) W. T. CARTER

3554. **Nuclear magnetic resonance spectroscopy.** R. Keuning (Unilever Res. Lab., Vlaardingen, Netherlands). *Chem. Weekbl.*, 1961, **57** (2), 13–18.—The principles of nuclear magnetic resonance spectroscopy are described, and some examples of its application are mentioned. (16 references.) M. J. MAURICE

3555. **Nuclear magnetic resonance and its application to the study of metals.** B. N. Finkel'shtein (I.V. Stalin Moscow Steel Inst.). *Zavod. Lab.*, 1961, **27** (1), 58–61.—The principles of nuclear magnetic resonance are reviewed, and applications to problems such as the study of ageing of duralumin, the diffusion coeff. of Li and Al, and the positions of protons in the lattice of ZrH_2 are discussed. G. S. SMITH

3556. **New technique for rapid determination of resonance positions of NMR spectra.** B. H. Arison and N. R. Trenner (Merck Sharp & Dohme Res. Lab., Div. of Merck & Co., Inc., Rahway, N.J., U.S.A.). *Anal. Chem.*, 1960, **32** (13), 1904.—Resonance positions are determined by projecting a suitable scale image on to the spectrum, adjusting to the proper size by lining up with an appropriate reference and side band, and reading the resonance positions directly. Excellent agreement is shown between this method and the normal method of interpolation. C. B. BAINES

3557. **Laboratory calibration of the neutron moisture meter.** P. G. Marais and W. B. de V. Smit (W. Province Fruit Res. Inst., Stellenbosch, S. Africa). *S. Afr. J. Agric. Sci.*, 1960, **3** (4), 581–601.—Factors affecting the performance of the neutron moisture meter (*Anal. Abstr.*, 1960, **7**, 5520) have been investigated with sand, soil and peat moss, all containing moisture in varying degrees from ovendryness to saturation. The sensitivity and shape of the calibration curve are dependent on the size of the calibration drum, the diameter of the access tube, and the position of the source relative to the counter. The resolution of the instrument is dependent on the moisture content of the volume "sampled," which is approx. a sphere, the radius of which increases from $\simeq 10$ cm at 100% moisture to >24 cm at moisture contents $<4\%$. Calibration curves obtained with peat moss and sugar - sand mixtures differed from each other and from the curves obtained with soils, and no single curve is suitable for all materials. G. SKIRROW

3558. **Accuracy in mass-spectrometric isotope analysis.** H. Birkenfeld (Inst. f. Physikalische Stofftrennung, Leipzig, Germany). *Kernenergie*, 1960, **3**, 906–910 (in German).—In the mass-spectrometric determination of the relative abundances of isotopes certain errors are possible, *e.g.*, by insufficient resolution, pre-separation in the ion source or the inlet system, memory effect or contaminated samples. The nature of these errors, the evaluation of their quant. influence, and the possibilities of their elimination in isotopic analysis are reported. NUCL. SCI. ABSTR.

3559. **Instrumental measurement of total ionisation in the mass spectrometer.** H. E. Lumpkin and J. O. Beauxis (Humble Oil and Refining Co., Baytown, Tex.). *Anal. Chem.*, 1960, **32** (13), 1815–1818.—Modifications to an analytical mass spectrometer are described which permit the direct measurement of a signal proportional to the total ionisation. This auxiliary equipment is simple to operate and accurate in use, and does not interfere with the normal operation of the instrument. The electrodes normally used to repel ions from an ionisation chamber are biased negatively with respect to the chamber body, and the total ionisation current collected is amplified and measured. Results for pure compounds indicate that the observed ratio of peak height to total ionisation is more reproducible than are replicate charges with a constant-vol. pipette. C. B. BAINES

See also Abstract—**3300**, Use of mass spectrometry in organic analysis.

ERRATA

February (1961) issue, abstract 449, line 2. *For* aesium *read* caesium.
Abstract 543, line 3. *For* C. H. Bheemasankara Rao *read* C. Bheemasankara Rao.
Abstract 545, line 3. *For* A. F. Korvezee *read* A. E. Korvezee.
Abstract 601, line 4. *For* R. T. Magee *read* R. J. Magee.

March (1961) issue, abstract 1037, line 4. *For* D. K. Banerjea *read* D. Banerjea.
Abstract 1336, line 2. *For* L. Halász *read* I. Halász.

April (1961) issue, abstract 1349, line 1. Insert **VIII.** after **standards.**
Abstract 1364, line 10. *For* Ca *read* Cu.
Abstract 1383, line 24. *For* and *read* or.
Abstract 1473, line 11. *For* cobaltniitrite *read* cobaltinitrite.

May (1961) issue, abstract 1835, line 3. *For* F. R. Sheyanov *read* F. R. Sheyanova.
Abstract 1876, line 2. *For* F. P. Sheyanova *read* F. R. Sheyanova.

July (1961) issue, abstract 2747, line 6 should read "described by which radio-elements, in trace amounts,"
Abstract 2822. The last term in the formula after line 16 should read $(1-e^{-\lambda\tau/\omega})$.

Now available in English Translation

RADIOCHEMISTRY U.S.S.R.

translated from the Russian RADIOKHIMIIA

under the editorship of

A. G. MADDOCK, *Cambridge*

SELECTED CONTENTS OF VOLUME I NO. I—JUST PUBLISHED

A. I. MOSKVIN, G. V. KHALTURIN and A. D. GEL'MAN: Investigation of the complexing of trivalent americium in oxalate and ethylenediamine tetra-acetate solutions by the ion-exchange method.

V. P. SHVEDOV and A. V. STEPANOV: An electromigration method of determination of the stability constants of complex compounds of elements present in micro-concentration—I. Determination of the stability constants of complex compounds of some lanthanides with the ethylenediamine tetra-acetic acid anion.

I. E. STARIK, A. D SHEIDINA and A. I. IL'MENKOVA: The state of micro-quantities of radioelements in dilute solutions—VII. Study of the state of proactinium in aqueous solution by the adsorption/desorption method.

V. I. SPITSYN and V. V. GROMOV: The problem of the effect of the radioactivity of precipitates on their sorption properties.

D. M. ZIV and V. A. ISHINA: Investigation of the electrochemical deposition of bismuth from its dilute solutions, by means of radioactive indicators I.

L. K. PONOMAREVA and V. L. ZOLOTAVIN: The determination of radioactive strontium in water from open reservoirs.

E. K. GERLING and Ia. A. SHUKOLIUKOV: The composition and content of xenon isotopes in uranium minerals.

K. A. PETRZHAK, E. B. NIKOL'SKAIA, Iu. G. PETROV and E. A. SHLIAMIN: The feasibility of applying to the study of fragmentary isotopes the technique of stopping and collecting fission fragments in a gas—I. Radiochemical examination of the disintegration of fragments using their range as a criterion.

A. N. MURIN and Yu. G. PLINER: The mechanism of formation of anomalous mixed crystals.

V. B. SHEVCHENKO, A. S. SOLOVKIN, I. V. SHILIN, L. M. KIRILLOV, A. V. RODIONOV and V. V. BALANDINA: The influence of the nature of the diluent on the extraction of uranyl nitrate by tributyl phosphate.

Two issues will be published per annum.

Subscription rates: £15 ($40.00) *per annum.*

Send for a free inspection copy

PERGAMON PRESS

OXFORD LONDON NEW YORK PARIS

Headington Hill Hall, Oxford. *4 & 5 Fitzroy Square, London, W.1*

122 East 55th Street, New York 22, N.Y.

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångström unit	Å
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
Colour Index	C.I.
concentrated	conc.
concentration	concn.
constant	const.
crystalline / crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c/s
density	ρ
density, relative	d or wt. per ml
dilute	dil.
direct current	d.c.
distilled	dist.
electromotive force	e.m.f.
equivalent	equiv.
ethylenediaminetetra-acetic acid	EDTA
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
hour	hr.
hydrygen ion exponent	pH
infra-red	i.r.
insoluble	insol.
international unit	i.u.
kilogram	kg
kilovolt	kV
kilowatt	kW
liquid	liq.
maxim-um, -a	max.
melting-point	m.p.
microgram	μg (not γ)
microlitre	μl
micromole	μmole
micron	μ
milliampere	mA
milli-equivalent	milli-equiv.
milligram	mg
millilitre	ml
millimetre	mm
millimicrogram	mμg
millimolar	mM
millivolt	mV
minute (time)	min.
molar (concentration)	M
molecul-e, -ar	mol.
normal (concentration)	N
optical rotation	α_L
ounce	oz
parts per million	p.p.m.
per cent.	%
per cent. (vol. in vol.)	% (v/v)
per cent. (wt. in vol.)	% (w/v)
per cent. (wt. in wt.)	% (w/w)
potential difference	p.d.
precipitate (as a noun)	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_λ^t
relative band speed	R_F
relative humidity	r.h.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_\lambda^t$
square centimetre	sq. cm
standard temp. and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	$\bar{v}$.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, *e.g.*, Fe^{II}, Mo^{V}. Substances in the ionic state are represented by Na^+, Fe^{3+}, Fe^{3+}, etc., for cations and by Cl^-, SO_4^{3-}, PO_4^{3-}, etc., for anions.

The chemical nomenclature used in the abstracts is that recommended by The Chemical Society ("Handbook for Chemical Society Authors", The Chemical Society, London, 1960), and may differ from that used in the original articles.

Volume 8. No. 8, Abstracts 3141 – 3559 August, 1961

ANALYTICAL ABSTRACTS

A PUBLICATION OF
THE SOCIETY FOR ANALYTICAL CHEMISTRY

EDITORIAL COMMITTEE

Chairman: B. A. Ellis

Members: B. S. Cooper, C. H. R. Gentry, C. A. Johnson, H. F. W. Kirkpatrick, B. J. Walby, W. A. Waygood, K. A. Williams
and
President of the Society: A. J. Amos

Hon. Secretary of the Society
R. E. Stuckey

Hon. Treasurer of the Society
D. T. Lewis

Editor: Norman Evers, B.Sc., Ph.D., F.R.I.C.

Assistant Editor: Mrs. H. I. Fisk, B.Sc.

CONTENTS

Abstract

General Analytical Chemistry
Reviews; reagents; methods of general application 3141

Inorganic Analysis
General methods; elements (in order of the Periodic Table); minerals; industrial products 3150

Organic Analysis
Elements and radicals; organic compounds; industrial products 3299

Biochemistry
BIOLOGICAL FLUIDS; ANIMAL AND VEGETABLE TISSUES 3389
PHARMACEUTICAL ANALYSIS 3443
FOOD
Food additives; beverages; edible oils and fats; vitamins 3466
SANITATION
Air; water; sewage; industrial wastes 3486
AGRICULTURAL ANALYSIS
Soil; fertilisers; herbicides; pesticides; animal feeding-stuffs 3497

General Technique and Apparatus
GENERAL 3502
CHROMATOGRAPHY; ION EXCHANGE; ELECTROPHORESIS 3508
OPTICAL 3525
ELECTRICAL 3540

Printed and published for the Society for Analytical Chemistry by W. Heffer & Sons Ltd., Cambridge, England.
Communications to be addressed to the Editor, Norman Evers, 14, Belgrave Square, London, S.W.1.
Enquiries about advertisements should be addressed to Walter Judd Ltd., 47, Gresham Street, London, E.C.2.
Entered as Second Class at New York, U.S.A., Post Office.